THE NICKEL BULLETIN

A Monthly Summary of Recent Literature

SEPTEMBER / OCTOBER

1959 Volume 32 No. 9-10

THE MOND NICKEL COMPANY LIMITED

THAMES HOUSE MILLBANK LONDON SW1

Telephone: VICtoria 3888

INDEX

Nickel Page

Falconbridge Nickel Mines, Ltd.: Review of Plant and Operations 279
Conversion of Nickel Oxide to Nickel 279
Optical Properties of Nickel 279
Solid Solubilities of Iron and Nickel in Beryllium 279
Gas Desorption from Nickel Powders 280
Magnetometric Titration of Nickel 280
Interaction of Methane and Nickel Oxide 280

Electrodeposition and Other Coating Methods

Automatic Addition of Chemicals to Nickel-Plating Solutions 281
Influence of Cathode-Film Thickness on Levelling 281
Avoidance of Roughness in Nickel Deposits 282
Orientation of Nickel Deposits 282
Detection of Porosity in Electrodeposited Nickel 282
Influence of Gas Bubbles on the Formation of Pores in Nickel Deposits 283
Nickel-Plating of Magnesium Alloys 283

Non-Ferrous Alloys

Mechanical Properties of Nickel-Copper Alloys 283
Constitutional Diagram of the Copper-Nickel-Manganese System 283

Nickel-Iron Alloys

Permanent-Magnet Materials 284
Magnetic-Properties/Temperature Relationships of Nickel-Iron Alloys 284
Ageing of Nickel-Iron-Titanium-base Spring Alloys 284
Brazing of Graphite to 'Nilo-K' 284

Cast Iron

Austenitic Corrosion-Resistant Nickel-Alloy Cast Iron 284
Wear-Resistant Cast Materials for Use in Crushing and Grinding Plant 285
Welding of S.G. Iron and Grey-Iron Castings 286

Constructional Steels

Influence of Metallurgical Variables on the Fatigue Properties of High-Strength Steel 286
Brittle Fracture Studies of Nickel-containing Steels 287
Low-Temperature Properties of Nickel-containing Steels 287
Nickel-containing Steels for Low-Temperature Applications in the Petroleum Industry 288
High-Expansion Nickel-Manganese Steel 288
Nickel-Chromium-Molybdenum High-Strength Steel Castings 288
Materials for High-Temperature Gear Applications 288
Development of Filler Wires for Welding High-Strength Steels 289

Heat- and Corrosion-Resisting Materials

Developments in Materials for High-Temperature Service 290
Vacuum-Melted Austenitic Steel 290
Precipitation-Hardening Stainless Steel 290
Nickel-Chromium-base High-Temperature Alloy 290
Solid-Solution, Carbide and Precipitation Hardening of Nickel-and Cobalt-base Alloys 290
Influence of Static Pre-Straining on the Fatigue Properties of Nickel-containing Steels at High Temperatures 292
Influence of Minor Alloying Additions on a Nickel-Chromium Alloy 292
Influence of the Degree of Stabilisation on Precipitation Reactions in Niobium-containing Austenitic Steels 292
Mechanical Properties of Structural Materials after Rapid Heating 293
Compressive, Bearing and Shear Creep-Rupture Properties at Elevated Temperatures 294
Tensile, Compressive, Bearing and Shear Properties of Sheet Steels at Elevated Temperatures 294

Heat- and Corrosion-Resisting Materials (*contd.*) Page

Effect of Cold-Working on Nickel-Chromium Alloys 295
High-Temperature Properties of 'Hastelloy B' and 'Hastelloy W' 295
Properties of 'Hastelloy B': Influence of Thermal Cycles Involved in Welding 296
Heat-Resistance of Welded Joints in a Nickel-Chromium-base Alloy 296
Protection of Molybdenum from Oxidation at High Temperatures 297
Thermal-Fatigue Cracking of Gas-Turbine Blading 297
Tubing for High-Temperature Fluid-Transport Service 297
Creep Testing of Steels Used in Steam Plant 298
Use of Austenitic Steels for Power Plant 298
Influence of Composition and Microstructure on Stress Corrosion of Precipitation-Hardenable Austenitic Steel 299
Spectral-Reflectivity and Solar-Absorptivity Data on Nickel-containing Materials 299
Corrosion of Condenser and Heat-Exchanger Tubes 299
Dislocation Interactions in Chromium-Nickel Stainless Steel 300
Stability of Austenitic Stainless Steels at Low Temperatures 301
Wear-Resistance of Austenitic Stainless Steel in Liquid Nitrogen 301
Reactor Material Specifications 302
Influence of Irradiation on the Properties of Stainless Steel 302
Report of the Corrosion Committee of B.I.S.R.A. 302
Developments in Materials Resistant to Atmospheric Corrosion 303
" " " " " Chemical Corrosion 303
Influence of Structural Factors on Corrosion of Stainless Steel 303
Intergranular Corrosion of Stainless Steels: Determination of Time/Temperature/Sensitisation Diagrams 304
Influence of Environment on Stress Corrosion of Stainless Steel 305
Stress Corrosion of Stainless Steel in Chloride Waters 305
Thermal-Cycling Stress-Corrosion Tests on Stainless Steel in Lithium-Hydroxide-containing Water 306
Corrosion of Chromium-Nickel-Molybdenum Stainless Steels in Phosphoric Acid 307
Corrosion of Nickel-containing Aluminium Alloys in High-Temperature Water 307
Corrosion of Austenitic Stainless Steels in High-Temperature Water Under Heat-Transfer Conditions 308
Corrosion of Nickel-containing Magnesium Alloys 308
Corrosion of Sour-Water Stripping Units 308
Corrosion-Resistance of Titanium-, Zirconium- and Nickel-containing Materials in Chemical-Plant Environments 309
Corrosion Tests on Nickel-containing Materials in Plant Processing Tall Oil 309
Technique for Investigation of Pitting Corrosion of Stainless Steel 309
Surface Finishing of Stainless Steels 310
Use of Nickel-Chromium-Boron Hard-Facing Alloys in the Sugar Industry 310
Hard-Facing with Nickel-base Alloys 310
Diffusion-Bonding Characteristics of 'Zircaloy-2' 311
Welding of Ferritic to Austenitic Steels: Review of Progress 311
Pressure-Welded Austenite/Ferrite Tube Junctions 312
Thermal-Shock Testing of Pressure-Welded Austenite/Ferrite Junctions 314
Stresses at the Joint Face of Austenite/Ferrite Welds 314
Welding of Dissimilar Metals with 'Inco-Weld A' Electrodes and Wires 314

Analysis 315

Patents 315

ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

NICKEL

Falconbridge Nickel Mines, Ltd.: Review of Plant and Operations

'The Falconbridge Story.'

Canadian Mining Jnl., 1959, vol. 80, June, pp. 102–230.

The whole of the June issue of the Canadian Mining Journal is devoted to a comprehensive technical review (prepared by staff of the Company) of the mining, sintering, smelting, and refining plants and operations of Falconbridge Nickel Mines, Ltd. Since its incorporation in 1928 the Company has increased its holdings in Sudbury, Canada, to many times the original acreage, and up to the end of 1958 has mined a total of 23,750,000 tons of ore, from which approximately 600,000,000 lbs. of nickel and 325,000,000 lbs. of copper have been refined. Its proven reserves are only slightly less than the total ore mined to date, and diamond drilling has indicated additional reserves of 20,000,000 tons.

The review, which is supplemented by flowsheets, numerous drawings, photographs and tabular data, is divided into sections concerned respectively with: Company history and organisation; geology; mining practice and description of individual mines (Falconbridge, East, McKim, Hardy, Boundary, Onaping, Fecunis Lake and Longvack mines); milling, smelting and refining; control laboratory; metallurgical research; mine engineering; work study; design and general engineering; maintenance; utilities; safety; accounting; purchasing and warehousing; property; industrial relations; and townsites.

Conversion of Nickel Oxide to Nickel

W. M. MAHAN, N. B. MELCHER, J. P. RIOTT and E. J. OSTROWSKI: 'Conversion of Nicaro Nickel Oxide to Nickel Metal.'

U.S. Bur. Mines, Report 5465, 1959; 36 pp.

Conversion of nickel oxide to metallic nickel by sintering and reduction in electric furnaces having proved uneconomical, the work described was carried out, under the aegis of the U.S. Bureau of Mines and the General Services Administration, with the aim of (1) determining the feasibility of using direct reduction and melting processes to convert the oxide produced at Nicaro, Cuba, to specification-grade ingot (i.e., containing a minimum of 98·5 per cent. of nickel plus cobalt, and a maximum of 1 per cent. of cobalt), and (2) to obtain data, in pilot-plant operation, which would permit estimation of the cost of producing 12,000,000 lbs. of ingot nickel per year.

Laboratory tests verified that, using ceramic saggers and a tunnel kiln, Nicaro nickel oxide could, in the presence of anthracite fines and dolomite, be reduced to sponge nickel, when heated at approximately 1900°F. (1035°C.) for from 2 to 4 hours. (The anthracite served as a reducing agent and the dolomite was introduced to control the sulphur content of the sponge nickel.) Specification-grade nickel ingots were then obtained by melting the sponge nickel in an open-hearth furnace.

Full details of the equipment and procedures used, data obtained and production costs are given in the report, in sections relating to: laboratory tests on conversion of nickel oxide to metal, pilot-plant production of sponge nickel, and pilot-plant production of nickel ingots from the sponge nickel.

Optical Properties of Nickel

S. ROBERTS: 'Optical Properties of Nickel and Tungsten and their Interpretation according to Drude's Formula.'

Physical Rev., 1959, vol. 114, Apr. 1, pp. 104–15.

The author reports new optical data for nickel at 88°, 298° and 473°K. (−185°, 25° and 200°C.) in the wavelength range 0·365 to 2·65 microns. It is shown that the data depend on the wavelength in a manner which is in good quantitative agreement with a formula initially proposed by DRUDE (*Physikalische Zeitsch.*, 1900, vol. 1, p. 161).

Mechanical Properties of Nickel

See abstract on p. 283.

Solid Solubilities of Iron and Nickel in Beryllium

S. H. GELLES, R. E. OGILVIE and A. R. KAUFMANN: 'The Solid Solubilities of Iron and Nickel in Beryllium.'

Trans. Metallurgical Soc., Amer. Inst. Mechanical Engineers, 1959, vol. 215, Aug., pp. 695–702.

The solid-solubility limits of iron in beryllium were determined, between 850°C. and 1200°C., by

analysis of differential-type multi-phase diffusion couples, using an X-ray-absorption technique. The maximum value of the solubility limit was found to be $0{\cdot}92\pm0{\cdot}02$ at. per cent. (5·46 wt. per cent.) at 1225°C., the eutectic temperature. The solubility of nickel in beryllium was established between 900°C. and 1200°C. The maximum solubility was $4{\cdot}93\pm0{\cdot}01$ at. per cent. (25·2 wt. per cent.) at 1065°C., the eutectoid temperature.

Diffusion-couple analysis revealed the existence in the beryllium-nickel system, at a composition of approximately 8 at. per cent. (36 wt. per cent.) nickel, of a previously unreported high-temperature phase, which decomposed eutectoidally at 1065°C. The presence of this phase was confirmed by thermal and metallographic analysis of the structure resulting from eutectoid decomposition.

Gas Desorption from Nickel Powders

J. F. WATSON: 'A Study of Gas Desorption of Nickel Powders.'

Dissertation Abstracts, 1958, vol. 19, p. 1334.
Metal Powder Report, 1959, vol 13, June, pp. 199–200.

A study was made of the gases desorbed from a variety of commercial nickel powders when heated to elevated temperatures. The results confirmed that the mechanisms of gas evolution and the types of gas evolved are dependent on the method of production of the nickel powder. Details are given of the nature and quantities of the gases found in the respective powders, and of the temperature-dependence of desorption.

Magnetometric Titration of Nickel

M. S. BLACKIE and V. GOLD: 'Magnetometric Titration: A New Method of Chemical Analysis.'

Nature, 1959, vol. 183, June 27, p. 1804.

It is known that the addition of paramagnetic solutes to water brings about a marked reduction in the nuclear spin-lattice relaxation time of solvent protons, the inverse of the relaxation time being linearly related to solute concentration according to the equation:

$$(T_1^{-1}—T_0^{-1}) = \alpha\mu N^2C$$

where T_1 and T_0 are spin-lattice relaxation times in the presence and absence of added solute, respectively; α is a proportionality constant; μN is a parameter expressing the effectiveness of a particular solute in this phenomenon, sometimes called the 'effective' paramagnetic moment of the solute; C is the concentration of the solute. In the case of solutions of paramagnetic complex ions, the magnitude of the effect (that is, the value of μN) depends on the nature of the co-ordinated groups. The authors find that the phenomenon enables the replacement of one ligand by another in solution to be followed with sufficient precision to form the basis for a method of determining the concentration of metal ions, and it is now suggested that this method of analysis should be widely applicable where the addition of a complexing agent to the solution of a paramagnetic ion in solution causes the formation of a new complex ion of different μN and of high stability in solution. The analyses can conveniently be carried out by use of a low-resolution nuclear magnetic-resonance spectrometer. A feature which makes the technique attractive in analytical chemistry is the small amount of solute required.

In this short communication the application of the method to titration of nickel(II) is illustrated.

Interaction of Methane and Nickel Oxide

S. BRANSOM and A. DANDY: 'The Interaction of Methane and Nickel Oxide.'

Trans. Faraday Soc., 1959, vol. 55, July, pp. 1195–9.

The experiments reported formed part of a study of the nickel oxides as oxidation catalysts. The aspects of interest were the lability of lattice oxygen in the oxidation of CH_4 to CO_2 and H_2O, the type and extent of CH_4 adsorption, and the effect, on adsorption and reaction, of various pretreatments of the oxide. The mechanism of adsorption on semi-conducting oxides is thought to be related to the mechanism of semi-conduction in the oxides, and it seemed useful therefore to use oxides whose semi-conductivities were know to differ. Nickel oxide is a *p*-type semi-conductor, and it has been shown that the addition of univalent ions increases the semi-conductivity, whilst the addition of trivalent ions reduces it. It was expected that such differences would be reflected in the adsorption and decomposition of a donor adsorbate such as CH_4.

Lithium and chromium were employed as the altervalent ions and were added to NiO as solutions of their nitrates. The mixture of nickel oxide and solution, calculated to produce 1 mole per cent. of foreign ion per mole of Ni^{2+} ion, was dried at 105°C. and annealed in oxygen-rich air for 4 hours at 1100°C. Un-doped nickel oxide was treated in the same way, in order to reproduce any surface changes which might occur. The interaction of CH_4 and the oxides was followed by the change of pressure with time in a constant-volume apparatus.

It was found that both the un-doped nickel oxide and the nickel oxide containing foreign ions were inert towards CH_4 at temperatures below 400°C. After partial reduction with CH at $t > 400$°C. the oxides were active from 200° to 450°C. Methane was adsorbed to a small extent on NiO and NiO–Cr_2O_3 at 200°–300°C., and to a relatively large extent on NiO–Li_2O. Maximum adsorption was observed at *ca.* 250°C., and the adsorption on NiO–Li_2O was reversible. The capacity of each oxide to absorb CH_4 decreased with progressive reduction of the oxide and finally disappeared. The activity of the reduction reaction leading to CO_2 and H_2O increased, with reduction of the NiO, to a reproducible maximum. It is shown that the addition of CO to the gas phase inhibits the reduction reaction, and the deceleration of the reaction with CH_4 is attributed to the accumulation, during the reaction, of irreversibly adsorbed CO.

ELECTRODEPOSITION AND OTHER COATING METHODS

Automatic Addition of Chemicals to Nickel-Plating Solutions

M. G. OSMAN: 'Automatic Additions of all Chemicals to the Nickel-Plating Baths.'

Plating, 1959, vol. 46, July, pp. 819–22.

This article opens with a description of the four steps involved in manufacturing gramophone records, as typified by a method generally used. The vital importance of correct electroforming is emphasised, as a factor in attaining optimum quality in the finished product.

In electroforming, a number of the problems associated with other uses of electrodeposition are intensified. Among them the question of stress in the deposit is particularly vital and in this connexion the purity of the electrolyte is of primary significance. It therefore becomes essential to control, at a very high level of accuracy, the quantity and quality of the replenishment media added to the bath.

The article describes, with detailed drawings and other illustrations, equipment devised for automatic and closely controlled addition of chemicals to nickel electroforming solutions. The respective units of the apparatus, working in conjunction with a water-level control, add all chemicals to the solution: anode and cathode are placed in the tank by conventional methods.

Experiments in the plating shops of the Radio Corporation of America have shown that use of this method of addition makes possible much closer regulation of the conditions in the solution, with resultant control of the internal stress, and of the physical properties of the deposit. Charts demonstrate the effects, on concentration of nickel sulphate and nickel chloride, and on pH, of manual *vs.* automatic addition.

Influence of Cathode-Film Thickness on Levelling

S. E. BEACOM: 'The Influence of Cathode-Film Thickness on Levelling.'

Plating, 1959, vol. 46, July, pp. 814–18.

Among the several hypotheses which have been advanced to account for levelling action in the electrodeposition of metals such as cobalt and nickel is one which takes into account the influence of the thickness of the cathode-film. This theory has been dealt with at length in papers by Foulke and Kardos, and other writers also have called attention to its significance.

It is pointed out by the present author that the assumption of a varying thickness of the cathode-film appears valid in the light of the proposal that levelling is initiated by an increase in polarisation on the peaks, which in turn is dependent upon a preferential adsorption of organic addition agents on these peaks. Thus, a thicker cathode-film in the valley of an irregular surface would slow down the diffusion of the addition agent to this region and therefore produce a polarisation somewhat less than that existing at the peak. This would lead to enhanced deposition of metal in the recessed region. (Reference is given to relevant literature.)

Consideration of a report by THOMAS (*Proc. Amer. Electroplaters' Soc.*, 1956, vol. 43, p. 60) indicates that, for a particular proprietary nickel-plating solution used by him, levelling was promoted by fall in temperature and increase in current density. The same investigator considered the effect of cathode-film thickness and found that the thickness of the film decreased with rise in temperature, and increased with increase in current density. His conclusions with regard to any possible difference in the cathode-film thickness in a dull-nickel (Watts-type) solution, as compared with that in a proprietary bright-nickel solution, were, however, inconclusive. The work now reported was carried out to obtain a clearer indication of the influence of the thickness of the cathode film on levelling of deposits made from two types of electrolyte.

The author notes the various methods which have been employed for measuring the thickness of the cathode film: his own experiments were made by the shadowgraph technique, based on the 'Schlieren' (shadowgraph) method employed by YEAGER (*Jnl. Accoustical Soc. Amer.*, 1953, vol. 25, p. 443). The principles of the method are outlined, and an illustrated description is given of the apparatus used.

Two plating solutions were used:

(1) A Watts-type solution containing nickel sulphate, nickel chloride, boric acid and a proprietary wetting agent.

(2) An electrolyte prepared from the same Watts-type basis solution, with the addition of two proprietary organic addition agents, one an alkyl sulphonate, the other a quaternary ammonium salt.

The starting pH of each solution was 3·0. Temperature of operation ranged from 80° to 160°F. (26·5° to 71°C.).

The conclusions summarised below are based on results given in a series of tables showing apparent film thickness *vs.* current density, apparent film thickness *vs.* temperature, apparent film thickness at flat and at valley on deposits made under various conditions, and increase in apparent film thickness from flat to valley, at a fixed current density. The values obtained justified the following conclusions:

(1) For both solutions the apparent cathode-film thickness is directly proportional to current density, and inversely proportional to temperature.

(2) The apparent cathode-film thickness in the valley is greater for solution (2) than for solution (1).

(3) The greatest apparent film thickness was found with solution (2) in the valley at the lower temperatures used and at high current density.

(4) At a given current density, the percentage increase in the apparent cathode-film thickness from

flat to valley for solution (2) was about twice the value for solution (1).

(5) The greater apparent cathode-film thickness formed in the valley in deposits from solution (2) may be related to the levelling ability of this electrolyte.

Commenting on these observations, the author emphasises that they refer only to specific plating solutions and that further work is essential before their general validity can be established.

Avoidance of Roughness in Nickel Deposits

D. R. NEWMAN: 'Smooth Copper and Nickel Deposits.' *Electroplating and Metal Finishing*, 1959, vol. 12, Aug., pp. 296–8.

Decorative copper and nickel electrodeposits are particularly susceptible to the roughening effect of insoluble particles present in the electrolyte. These particles are introduced into the solution in four ways: *via* the components being plated; as air-borne dust; as a result of some chemical or physical reaction in the solution itself (examples are listed), or by dissolution of the anode. The introduction of particles from the first three sources can be prevented relatively easily, but those originating in the anode are more difficult to control. The method generally employed is to enclose the anode in a bag, a procedure which, the author indicates, has its disadvantages.

In attempting to eliminate roughening in the deposits, particularly those produced from air-agitated solutions on articles having large surface areas, the author has considered the use of, and in some cases has adopted, diaphragm tanks in place of anode bags. In this article he describes the type of diaphragm construction which experience has shown to eliminate roughness in nickel and copper deposits.

Orientation of Nickel Deposits

B. C. BANNERJEE and A. GOSWAMI: 'Effects of Chloride on the Orientation of Nickel Deposits.' *Jnl. Electrochemical Soc.*, 1959, vol. 106, July, pp. 590–2.

The authors' previous work on the crystal structure and orientation of nickel electrodeposits had shown that the out-growth condition of nickel deposition in the presence of impurities can be modified to lateral growth by addition of sodium chloride. Since the chloride ion is known to increase the conductivity of the solution and to facilitate electron-transfer processes at the cathode, it was deemed of value to study its effects on the structure of nickel deposited from impure solutions, and so to gain an insight into the process of cathodic crystal growth.

Nickel was deposited, at constant current density and temperature (from solutions containing $NiSO_4.7H_2O$ 280, H_3BO_3 31, g./L.) on brass substrates, in the presence of from 0 to 100 g./L. of sodium chloride and varying amounts of aluminium sulphate, chromium sulphate, chromic acid, benzaldehyde, nitrobenzene, glycine, gelatine, benzene disulphonate, and 1 : 3 : 6 sodium salt of naphthalene trisulphonate. Deposition was carried out to a stage well beyond that at which polycrystalline deposits developed preferred orientations characteristic of electrolyte conditions. The pH of the solution was adjusted so that the deposits were of lateral-growth type in the absence of impurities. After each addition of impurities or of sodium chloride the orientation of the deposit was determined by electron-diffraction techniques.

The data presented indicate that sodium chloride suppresses completely, or at least minimises, the effect of such factors as: adsorption of compounds at the cathode, film formation in the catholyte which hinders the supply of ions and the electron-transfer process, and the formation of complex ions which contribute to out-growth deposition. The results suggest that chloride ions facilitate the desorption process of the surface-active compounds at the cathode surface and render more sites available for deposition, thus reducing the current density and contributing to lateral growth.

Detection of Porosity in Electrodeposited Nickel

F. OGBURN and D. W. ERNST: 'The Nature, Causes and Effects of the Porosity in Electrodeposits. III. Microscopic Detection of Porosity.' *Plating*, 1959, vol. 46, July, pp. 831-3.

The paper is one of a series reporting investigations carried out under the aegis of the *American Electroplaters' Society*. (For abstracts of parts 1 and 2 see Nickel Bulletin, 1954, vol. 27, No. 4, pp. 66–7; *ibid.*, 1957, vol. 30, No. 4, p. 55.)

It is pointed out that although detection of porosity in plated coatings has received much attention, the potential value of microscopical investigation has not been recognised. For example, the statement has been made that 'in hundreds of metallographic cross-sections of nickel deposits examined over a course of years, at magnifications from 100 to 500 diameters, not one pore had been encountered which could be recognized as such'.

The authors suggest that the reason for such failure is that most pores in nickel deposits are of less than 0·0001-in. (0·0025-mm.) diameter, and that the chances of sectioning such a small tube parallel to its axis must be very slight. If it is not sectioned parallel to the axis only a dot or oval would be seen, and even if the sectioning has been successfully performed, there is the possibility that the contours may be distorted by polishing or etching. It was realized, however, that a section perpendicular to the axis of the pore, parallel to the basis material, would be much more likely to cut a pore, and that a series of such cuts should make it possible to follow a pore from top to bottom. The paper reports successful results obtained by adoption of this technique.

The coating is polished parallel to the basis material and a pore then appears as a small dot. If it is larger than 0·05 mil (1·2 μ) in diameter it can be easily seen at ×400 magnification.

It is possible to confuse a pore which appears as a dot with surface defects introduced during polishing,

but such confusion may be minimised by polishing away another layer of metal and re-examining the same area, when the dot, if it is a pore, will appear at the same location. If a dot appears in two or more successive parallel sections, it can be considered to be a real discontinuity in the coating. A figure in the paper shows a series of photomicrographs taken of an identical area at four stages during parallel sectioning of a Watts nickel deposit.

Mounting of foils or thin flat specimens for parallel sectioning requires a technique somewhat different from that used for cross-sectioning. Details are given of the procedure found suitable for anchoring thin specimens and for mounting of attached coatings. Polishing and etching methods are described, as applied to nickel and nickel/chromium coatings.

The types of pore or defect found in the Watts nickel coating are pits (dead-end pores), cavities (enclosed pores), continuous pores, and bridged pores. The respective types are illustrated by a schematic diagram and by reference to the photomicrographs. As the observations made to date are somewhat limited, it is not possible at present to indicate the relative frequency of each type of pore, but it may be tentatively concluded that they are about equally prevalent.

It is suggested that in addition to its value for determination of porosity, the method of successive parallel sectioning may be useful for investigation of damage done to coatings by atmospheric corrosion.

See also

Influence of Gas Bubbles on the Formation of Pores in Nickel Deposits

F. OGBURN and D. W. ERNST: 'A.E.S. Research Project No. 13. The Nature, Cause and Effect of the Porosity in Electrodeposits. IV. Influence of Gas Bubbles on the Formation of Pores.'

Plating, 1959, vol. 46, Aug., pp. 957–8.

The suggestion that surface gas bubbles contribute to the formation of pores in electrodeposits has been rejected on several occasions. A further study of the influence of gas bubbles in this respect is described in the present paper.

In the study use was made of a cell which permitted the authors to observe deposition of nickel on the periphery of the cathode, i.e., which permitted observation of the growth of a simulated 'cross-section' of the nickel deposit during plating. When the cell was operated at low current density the deposit grew thicker at a uniform rate, with few visible defects. At high current densities, hydrogen was evolved in great excess, and the deposit became rough and had poor adherence. The procedure followed by the authors (to increase the current density until hydrogen was evolved and then to decrease it slightly) permitted hydrogen bubbles to remain attached to the nickel surface.

A series of photomicrographs are presented illustrating the formation of a void due to deposition of nickel over a surface gas bubble. It is concluded that the relative rates of hydrogen discharge and nickel deposition are the factors which determine whether a bubble is covered by the deposit or enlarges itself. When a gas bubble is large enough to break away from the nickel deposit it does so in such a manner as to leave gas in the pore formed by the deposit growing round the bubble, and so effectively prevents electrolyte from entering the pore and deposition from occurring. Although most of the pores observed were larger (10 mils) than commonly observed on plated components, smaller (1-mil) pores were also observed. The experimental procedures employed differ greatly from typical plating procedures in several respects; however, the observations reported may, it is considered, indicate a general mechanism of pore growth.

Nickel-Plating of Magnesium Alloys

V. I. LAINER and G. S. KONSTANTINOVA: 'Copper and Nickel Plating of Magnesium Alloys.'

Vestnik Mashinostroeniya, 1959, vol. 39, No. 2, pp. 62–5.

Details are given of a technique employed for preparation of the magnesium alloys, immersion coating with zinc, electroplating with copper, and finally coating with nickel and chromium by conventional electrodeposition practice.

For details see *Chemical Abstracts*, 1959, vol. 53, June 10, p. 9850.

NON-FERROUS ALLOYS

Mechanical Properties of Nickel and Nickel-Copper Alloys

V. A. PAVLOV and I. A. PEVETURINA: 'Mechanical Properties of Alloys of Nickel and Copper.'

Fiz. Metal. i Metalloved., Akad. Nauk S.S.S.R., 1958, vol. 6, pp. 717–24.

Report of investigation of the effect of temperature of working and rate of deformation on the mechanical properties of nickel and of nickel alloys containing 10, 20, 40 and 60 per cent. of copper. The study was made over the temperature range $-196°$ to $+700°C$.

The results are summarised in *Chemical Abstracts*, 1959, vol. 53, June 10, pp. 9978–9.

Constitutional Diagram of the Copper-Nickel-Manganese System

B.-C. CHANG: 'Copper-Nickel-Manganese Alloy.'

Isvest. Vysshikh Ucheb. Zavedenii, Tsvetnaya Met., 1958, No. 5, pp. 107–15.

A ternary constitutional diagram was constructed on the basis of hardness tests on, and X-ray, chemical and microscopic analyses of, forty-one alloys containing up to 35 per cent. manganese, up to 35 per cent. nickel, remainder copper. The salient features of the diagram are noted in *Chemical Abstracts*, 1959, vol. 53, July 10, p. 12134.

Hard-Facing with Nickel-base Alloys

See abstract on p. 310.

Use of Nickel-Chromium-Boron Hard-Facing Alloys in the Sugar Industry

See abstract on p. 310.

NICKEL-IRON ALLOYS

Permanent-Magnet Materials

R. J. FABIAN: 'Permanent-Magnet Materials.'

Materials in Design Engineering, 1959, vol. 50, July, pp. 108–14.

In this article the author reviews the various groups of materials which have been developed for use as permanent magnets, and, in brief notes on the individual materials comprising these groups, draws attention to the advantages and the range of properties they offer, and to the applications for which they are suited.

Five groups of materials are discussed: (1) Magnet steels (carbon, tungsten, chromium and cobalt steels); (2) Iron-nickel-aluminium alloys of the 'Alnico' series (I, II, III, IV, V, VI and XII); (3) Magnet alloys designed to satisfy specialised needs, e.g., ease of fabrication, low-cost production, light weight; (4) Ceramic materials; (5) Fine-particle materials.

Where the information is available, data are tabulated indicating the nominal composition and the magnetic, physical, and tensile properties of each of the materials considered in the review.

Magnetic-Properties/Temperature Relationships of Nickel-Iron Alloys

J. J. CLARK and J. F. FRITZ: 'The Effect of Temperature on the Magnetic Properties of Nickel-Iron Alloys.'

Wright Air Development Center, Tech. Note 57-434, Dec. 1957; 33 pp.

J. J. CLARK and J. F. FRITZ: 'The Effect of Temperature on the A-C Magnetic Properties of Nickel-Iron Alloys.'

Ibid., Tech. Note 58-277, Aug. 1958; 40 pp.

The two reports record the results of a comprehensive investigation of the influence of temperature on the magnetic properties of the following nickel-iron alloys: 'Hipernik', 'Hipernik V', 'Deltamax', 'Hymu-80', 'Supermalloy', and 4–79 Mo-'Permalloy'.

Tech. Note 57-434 is concerned with the d-c magnetic properties of the six alloys. The curves obtained have been published in *Electrical Manufacturing*, 1958, vol. 62, Nov., pp. 135–9: the pertinent abstract in *Nickel Bulletin*, 1959, vol. 32, No. 3, pp. 80–1 gives particulars of test conditions and the compositions of the alloys.

The information contained in *Tech. Note* 58-277 relates to a-c properties measured at frequencies of 60, 400 and 1000 cycles over the temperature range −60°C. to +250°C. The results for each alloy are reported in the form of six curves (one for each of the three test frequencies) indicating, as a function of temperature, variation of (1) core-loss per unit of weight, and (2) rms volt-amperes per unit of weight.

The two reports, used in conjunction, provide basic design data for the temperature range studied.

Ageing of Nickel-Iron-Titanium-base Spring Alloys

M. M. BORODKINA: 'Ageing of Nickel-Iron-Titanium-base Spring Alloys.'

Fiz. Metal. i Metalloved., Akad. Nauk S.S.S.R., 1958, vol. 6, pp. 700–5.

Report of study of structural transformations in iron-base alloys containing nickel 30–42, titanium 0–6, per cent., and of the effect of chromium and aluminium on their ageing characteristics. Mechanical and magnetic properties were investigated and metallographic and X-ray-diffraction examination was carried out to establish the various phase relations of the iron-nickel-titanium system in the region investigated.

The observations made are summarised in *Chemical Abstracts*, 1959, vol. 53, June 10, p. 9972.

Brazing of Graphite to 'Nilo-K'

B. STAPLETON: 'Brazing Graphite to 'Nilo-K' Tubes.'

U.K. Atomic Energy Authority, Industrial Group, Report 140 (*RD/CA*), 1959; 5 pp. + figures.

Obtainable from H.M. Stationery Office. Price 2/6.

The report gives details of a technique which, developed at the Authority's Research and Development Branch, Capenhurst, as a means of brazing graphite samples to carrier tubes fabricated from the iron-nickel-cobalt alloy 'Nilo-K', produced leak-tight joints which permitted determination of the permeability of the graphite at high pressures and at temperatures up to 900°C.

The method, essentially a vacuum-brazing technique, involves assembling the specimen and tube, with the brazing wire wrapped in position, inside a silver envelope. The envelope is then evacuated, the assembly is hot-degassed, and finally the temperature is raised to effect the braze. Use was made of a copper-silver eutectic brazing wire with a 12 per cent. titanium core. The difficulties encountered during the initial attempts at brazing are described in the report, and particulars are given of furnace design and of the brazing technique finally evolved.

CAST IRON

Austenitic Corrosion-Resistant Nickel-Alloy Cast Iron

SOC. AUTOMOTIVE ENGINEERS: 'Alloy Iron Castings, Sand, Corrosion-Resistant.'

A.M.S. Specification 5392*F*, June 15, 1959.

Covers parts requiring moderate resistance to corrosion and heat at operating temperatures up to 800°F. (427°C.).

Compositional limits are as follows: carbon 2·4–2·8,

silicon 1·5–2·5, manganese 1·0–1·5, sulphur 0·12 max., phosphorus 0·30 max., chromium 1·8–2·4, nickel 14·0–16·0, copper 6·0–7·0, lead 0·003 max., per cent. If the castings have sections less than $\frac{3}{4}$ in. (18 mm.) thick, carbon and silicon may each be as high as 3·0 per cent. The form of tensile and of chemical-analysis specimens is laid down, and the tensile and hardness requirements for castings and/or test specimens are as shown below.

U.T.S.	.. ..	30,000 p.s.i. min. 13·5 t.s.i. min. 21·0 kg./mm.^{2} min.
Hardness	.. ..	140–180 (or Rockwell B 75–88)

It is required that parts made from this material shall be capable of being cooled to −75°F. (−60°C.) without the austenite transforming to martensite.

Wear-Resistant Cast Materials for Use in Crushing and Grinding Plant

T. E. NORMAN and G. E. OTT: 'Recent Developments in Wear-Resistant Cast Materials for Use in Crushing and Grinding Plant.'

Giesserei, 1959, vol. 46, July 30, pp. 441–7; Aug. 13, pp. 471–8.

The development of wear-resistant materials capable of extending the life of components used in crushing and grinding plant is a matter of fundamental interest to the ore-refining industry. The Climax Molybdenum Company have, at their plant in Climax, Colorado, carried out, over a considerable number of years, a comprehensive series of service tests to evaluate the wear-resistance of cast irons and steels developed with this aim in view. The Company processes over 30,000 tons of ore per day, and its plant therefore offered a means of carrying out long-time tests on a variety of materials under comparative conditions. It was possible, for example, within the scope of the programme, to evaluate, over a period of 20 years, the performance of 500 sets of ball-mill linings.

This paper presents the findings from tests on the following cast materials:

(1) An austenitic manganese steel (manganese 12, molybdenum 2, per cent.) containing, as a result of suitable heat-treatment, a fine distribution of carbides which contributes considerably to wear-resistance.

(2) Pearlitic and martensitic chromium-molybdenum steels containing up to 7 per cent. of chromium and 1 per cent. of molybdenum.

(3) A white cast iron containing chromium 15, molybdenum 3, per cent.

(4) A white cast iron containing nickel 3, chromium 2, molybdenum 1, per cent.

In the first part of the paper each of these materials is discussed, in some detail, in terms of composition, melting and casting, moulds and shrinkage, mechanical properties, machinability and weldability.

The second part of the paper comprises a review of the performance of these four groups of materials in relation to the service conditions under which they were evaluated, i.e., in applications involving exposure to 'notch wear' (Kerbverschleiss), 'mill wear' (Mahlverschleiss) or 'erosion'. 'Notch wear' occurs as a result of impact with large pieces of ore, which tear away relatively large particles of the structural material. 'Mill wear' is encountered when abrasive materials are ground between two wear-resistant materials: characteristic of this type is the wear suffered by the linings and grinding balls of ball mills. 'Erosion' is defined as the effects, on a wear-resistant component, of contact with very small, relatively free-moving abrasive particles. Rates of wear exhibited by parts used in applications involving exposure to each of these three types of wear are tabulated.

The authors draw attention to the factors which govern the selection of a wear-resistant material for a given application, and finally (using resistance to notch wear, to mill wear, or to erosion as a criterion) review the applications for which (as indicated by actual service results) the four cast materials are suitable. The findings are considered to be of interest not only to the ore-refining industry, but also to others (e.g., the coal, cement, and paper industries) in which comparable applications exist.

The paper includes a table (see below) which indicates the relative suitability of the four materials for service under each of the abrasive conditions.

Evaluation of Wear-Resistant Cast Materials for Grinding Plant

Type of Wear Involved During Service	'Notch Wear'	'Mill Wear'	'Erosion'
Descending Order of Suitability of the Materials Tested	15-3 Cr-Mo Cast Iron	15-3 Cr-Mo Cast Iron	15-3 Cr-Mo Cast Iron
	3-2-1 Ni-Cr-Mo Cast Iron	Martensitic Cr-Mo Steel	3-2-1 Ni-Cr-Mo Cast Iron
	Martensitic Cr-Mo Steel	3-2-1 Ni-Cr-Mo Cast Iron	Martensitic Cr-Mo Steel
	12-2 Mn-Mo Steel	Pearlitic Cr-Mo Steel	Pearlitic Cr-Mo Steel
	Pearlitic Cr-Mo Steel	12-2 Mn-Mo Steel	12-2 Mn-Mo Steel

Welding of S.G. Iron and Grey-Iron Castings

B. TOWNSHEND and E. O. PORTER: 'Process Welding of Nodular and Gray-Iron Castings.'

Welding Jnl., 1959, vol. 38, Aug., pp. 329s-34s.

In general, the procedures suggested as most suitable for use in welding S.G. iron or grey-iron castings either involve technical difficulties (e.g., maintenance of high preheat temperatures during welding) or suffer from economic drawbacks. The investigation described by the authors was therefore carried out in an attempt to determine a welding procedure which, under normal production-welding conditions, would (1) produce, in such castings, joints having mechanical properties comparable to those of the basis metal, (2) be economically practicable, and (3) be suited to the ability of the average qualified welder.

It is emphasised that the welding characteristics of the irons studied in the investigation are not necessarily representative of, and hence that the conclusions drawn are not necessarily applicable to, other grades of iron. (For details of the two grades of iron used see table in next column.) Welds were produced in cast test blocks designed to offer a high and consistent degree of restraint. Joint efficiency was determined by tensile tests on cross-weld specimens.

In exploratory tests the grey cast iron was welded with different types of electrode (the compositions of which are noted), using the shielded metal-arc, gas-shielded tungsten-arc, and oxyacetylene processes and a preheat and interpass temperature of 600°F. (315°C.). Cracking occurred in all the welds with the exception of those produced (by means of the shielded metal-arc process) with a 55-45 nickel-iron flux-coated electrode or (by oxyacetylene welding) with cast-iron or bronze filler metal. As a result of these findings, work in the main test programme was concentrated on development of a suitable procedure for shielded metal-arc welding of the cast irons, using the nickel-iron electrode.

The influence of the following variables was studied:

(1) *Pre-heat temperature at which the weld was made.*

(2) *The intermediate heat-treatment applied to buttered joints.*

The joints were 'buttered' by deposition of a single pass of weld metal, allowed to cool to room temperature, and then subjected to a stress-relief or annealing heat-treatment, following which the sample was welded to completion, either at room temperature or at various preheat temperatures.

(3) *The operational technique used in welding the irons by the shielded metal-arc process.*

The effects of varying the above three factors are discussed in relation to data tabulated on the tensile properties of the joint produced.

The authors conclude that the two grades of cast iron can be successfully welded by use of the shielded metal-arc process in conjunction with 55-45 nickel-iron electrodes and a preheat and interpass temperature of 600°F. (315°C.). When the surfaces of the joint are buttered with the nickel-iron electrodes at this preheat temperature and then subjected to stress-relief treatment at 1200°F. (650°C.) or to a full anneal, process welding of the remainder of the joint may be carried out at room temperature. Provided that care is taken to prevent the occurrence of weld porosity, the joints produced by this procedure are of a strength comparable to that of the basis metal. Weld porosity was minimised by use of a stringer-bead technique with which an arc length of about $\frac{3}{16}$-in. (0·5-cm.) was maintained.

S.G. and Grey Cast Irons Used in Welding Programme

Material Specifications:	Class 30 Grey Cast Iron ASTM A 278-53	60-40-15 Nodular Cast Iron ASTM A 395-56T
Condition before welding:	Stress-relief annealed	Ferritise annealed
Chemical analysis, per cent.		
Tc	3·02	3·55
Cc	0·72	0·28
Si	2·20	2·37
Mn	0·75	0·60
P	0·11	0·033
S	0·11	0·010
Graphite-flake condition	Uniform distributions, random orientation	
Matrix:	Pearlitic-lamellar and spheroidised	Ferrite, approximately 15 per cent. pearlite
Average properties:		
Ultimate tensile strength	37,000 p.s.i. 16·5 t.s.i. 26 kg./mm.²	68,000 p.s.i. 30·5 t.s.i. 49 kg./mm.²
Yield strength	—	31,000 p.s.i. 14 t.s.i. 22 kg./mm.²
Elongation, per cent.	—	17
Brinell hardness	187-205	180 max.

CONSTRUCTIONAL STEELS

Influence of Metallurgical Variables on the Fatigue Properties of High-Strength Steel

J. I. FISHER and J. P. SHEEHAN: 'The Effect of Metallurgical Variables on the Fatigue Properties of A.I.S.I. 4340 Steel Heat-Treated in the Tensile Strength Range 260,000-310,000 p.s.i.'

Wright Air Development Center, Tech. Report 58-289, Feb. 1959; 144 pp.

The investigation reported was carried out with the aim of determining which of the factors subject

to metallurgical control influence the fatigue properties of high-strength steel.

The material selected for investigation was a nickel-chromium-molybdenum steel (A.I.S.I. 4340 grade) heat-treated to tensile strengths in the range 260,000–310,000 p.s.i. (116–139 t.s.i.; 138–218 kg./mm.^{2}). The metallurgical variables studied related mainly to melting procedure, but a few tests were carried out to throw light on the effects of heat-treatment, retained austenite, and dissolved hydrogen and nitrogen.

In view of the growing belief that the size, type, concentration and location of non-metallic inclusions considerably influence the fatigue endurance of hardened steels, melting procedures were selected which would influence such inclusions. To provide a basis for comparison, a commercially produced heat of aircraft-quality 4340 steel was first fully tested and then arc-remelted in an argon atmosphere by the consumable-electrode process and re-tested. In addition, tests were carried out on heats of steel which had been induction-melted or arc-melted *in vacuo*, and on a series of heats which were induction-melted in air, 'blocked' with carbon instead of silicon, and deoxidised with carbon, titanium, aluminium or zirconium to obtain pronounced differences in the types of inclusion present. An estimate of the endurance limit of the specimens was obtained by means of the Prot method.

It was found, from study of the size of the non-metallic inclusions in each heat, that a correlation existed between the mean width of the largest inclusions and the ratio of endurance limit to ultimate tensile strength; the smaller the mean diameter of the inclusions, the higher the endurance-limit/tensile-strength ratio. Reduction in the amount of retained austenite and dissolved hydrogen and nitrogen present had no effect on the fatigue properties, possibly because of the overriding influence of large inclusions in the heat tested.

Brittle Fracture Studies of Nickel-containing Steels

G. M. ORNER: 'Effect of Specimen Geometry on Charpy Low-Blow Transition Temperature.'

Welding Jnl., 1959, vol. 38, Aug., pp. 315s–23s.

The study reported represents a further phase in the evaluation of the Charpy low-blow technique, a variation in the standard procedure which permits separation of the crack-initiation and the crack-propagation stages of the fracture process. Previous work by the author, carried out with the aim of extending the usefulness of the Charpy impact test, had demonstrated that, with respect to a pearlitic 3·3 per cent. nickel steel, the Charpy low-blow transition temperature was independent of notch and specimen geometry and was a property of the material tested (see, for example, abstract in *Nickel Bulletin*, 1959, vol. 32, No. 8, p. 259). The object of the present investigation was to determine (1) whether low-blow transition temperatures would be independent of notch and specimen geometry in a variety of steels, and (2) whether the low-blow technique could be even further simplified by striking the crack-initiating blow at a single convenient temperature, instead of at the various temperatures used for the crack-propagating blow.

Tests were conducted on a pearlitic 3·3 per cent. nickel steel, a carbon steel in the as-rolled condition, and a low-alloy nickel-chromium-molybdenum steel in the quenched-and-tempered condition. From the data obtained (which are presented in detail in the paper) the author concludes that modification of the testing technique permitting the crack initiating blow to be carried out at room temperature has no measurable effect on the temperature at which the low-blow transition occurs, and that the latter temperature is independent, within the limits of acuities and specimen sizes tested, of notch and specimen geometry.

Tensile, Compressive, Bearing and Shear Properties of Sheet Steels at Elevated Temperatures

See abstract on p. 294.

Low-Temperature Properties of Nickel-containing Materials

R. L. MCGEE, J. E. CAMPBELL, R. L. CARLSON and G. K. MANNING: 'Mechanical Properties of Certain Aircraft Structural Metals at Very Low Temperatures.'

Wright Air Development Center, Tech. Report 58-386, Nov. 1958; 57 pp.

Little information is available on the mechanical properties of aircraft structural materials at, and below, the temperature of liquid nitrogen. In view, therefore, of recent developments in the aeronautical field which involve the handling of liquid propellants at very low temperatures, the study reported was carried out to determine the tensile properties, at room temperature, −78°, −196° and −253°C., of nine structural materials of interest in this respect.

The following materials were tested: '17-7 PH' and 'AM-350' precipitation-hardenable stainless steels; low-alloy nickel-chromium-molybdenum steel (A.I.S.I. 4340); 'Tricent' nickel-chromium-molybdenum-silicon steel; three titanium alloys; an aluminium alloy; and a magnesium alloy. Data were obtained on modulus of elasticity, yield strength (0·2 per cent. offset), tensile strength, breaking strength, elongation, reduction-in-area, and notch strength.

Tensile strengths and moduli of elasticity of all the materials tended to increase as the test temperature was lowered. The 'Tricent' steel and one of the titanium alloys were, however, very brittle at −253°C. and failed in the fillets at lower stress levels than those obtained at −196°C. The yield strengths of all the materials also increased as the temperature was lowered, but at −253°C. only the '17-7 PH' stainless steel, the aluminium and magnesium alloys, and the other titanium alloys reached 0·2 per cent. offset before rupture. The yield-strength/density ratios exhibited by the titanium

alloys at the lower test temperatures were considerably higher than those of the other materials. The hardness of all the materials increased with decrease in temperature, and the notched impact-strengths of the 'Tricent' and A.I.S.I. 4340 steels, and of the aluminium and magnesium alloys, were reduced. At −253°C. notched impact energies were relatively low.

Nickel-containing Steels for Low-Temperature Applications in the Petroleum Industry

E. T. GILL and G. L. SWALES: 'Nickel-containing Steels for Low-Temperature Applications in the Petroleum Industry.'

MOND NICKEL CO., LTD., *Publn.* 1790.*
Reprint from *Brit. Petroleum Equipment News*, 1959, vol. 7, Spring, pp. 60–4.

See abstract in *Nickel Bulletin*, 1959, vol. 32, No. 6, p. 196.

High-Expansion Nickel-Manganese Steel

SOC. AUTOMOTIVE ENGINEERS: 'Steel, High-Expansion.'
A.M.S. Specification 5625*A*, June 15, 1959.

The specification governs cold-drawn bar, primarily for use in bolts and screws requiring a coefficient of expansion approaching that of aluminium alloys.

Compositional limits are as follows: carbon 0·55–0·65, silicon 1·00 max., manganese 5·00–6·00, sulphur 0·030 max., phosphorus 0·040 max., nickel 8·50–10·50, per cent. Tensile properties and hardness are laid down and the material is required to show a coefficient of thermal expansion not lower than $11{\cdot}5\times10^{-6}$ per degree F. over the temperature range 72°–600°F. (22°–315°C.).

A related specification (*A.M.S.* 5623, of even date) cover bars, forgings and forging stock of the same composition, showing the same coefficient of thermal expansion.

Nickel-Chromium-Molybdenum High-Strength Steel Castings

K. D. HOLMES, J. ZOTOS and P. J. AHEARN: 'High-Strength-Steel Castings: Mechanical Properties and Processing Techniques.'

Modern Castings, 1959, vol. 36, July, pp. 65–70.

The need for materials exhibiting a high strength/weight ratio has aroused considerable interest in the feasibility of increasing, without serious loss in ductility and toughness, the yield strength of high-strength cast steels from the present level of about 150,000 p.s.i. (67 t.s.i.; 105·5 kg./mm.²) to the region of 200,000 p.s.i. (89 t.s.i.; 140 kg./mm.²). In this paper the authors first review the factors involved in the production of such steels and then present data which illustrate the influence of composition, melting practice and heat-treatment in determining the degree to which the desired combination of ductility and high strength can be obtained.

The introductory section covers current practice with respect to refractories and raw materials, melting, pouring, heat-treatment and testing.

The main section is concerned with discussion of data derived from tensile and impact tests on castings from five heats of a low-alloy nickel-chromium-molybdenum steel, and from one heat of a low-alloy nickel-chromium-molybdenum steel containing 1·5 per cent. silicon (selected on account of its resistance to temper-embrittlement). Two heats of the former steel (one of which was used in the production of drum-cradle castings) and that of the latter were melted in an induction furnace; the remaining three heats were melted in an arc furnace. All the heats, with the exception of that mentioned above, were cast into finned blocks which permitted mechanical tests to be carried out on specimens representative of the thin fin, the midwall and centre sections of the casting. Castings from the arc-furnace melts were subjected both to low-strength and high-strength heat-treatments; castings in the other three heats were each subjected to a different heat-treatment schedule. Full details are given of the five heat-treatments employed and of the data derived from tests on specimens from each casting. Tensile strengths ranged from 140,000 to 270,000 p.s.i. (62·5 to 120 t.s.i.; 98·5 to 190 kg./mm.²).

In the authors' discussion of the results differences in melting and heat-treatment procedure are correlated with the mechanical properties obtained in the castings. This correlation emphasises the advantages of arc-furnace melting: specimens taken from the fins of one test casting combined a tensile strength of 225,000 p.s.i. (100·5 t.s.i.; 158 kg./mm.²) with a 35 per cent. reduction-in-area and an impact strength at −40°F. (−40°C.) of 15 ft. lb. (2·07 kgm.). The mechanical properties of the castings heat-treated to high-strength levels were found to decrease with increase in section size.

Materials for High-Temperature Gear Applications

E. G. JACKSON, C. F. MUENCH, E. H. ROWE and E. H. SCOTT: 'Evaluation of Alloys for High-Temperature Gear Applications.'

Wright Air Development Center, Tech. Report 58-546, Dec. 1958; 79 pp.

The test programme described in the report was carried out with the aim of evaluating commercially available materials in relation to their suitability for use in gears operating at temperatures up to 700°F. (370°C.) in high-speed aircraft.

Eight materials were selected for testing: (1) 'M50' Cr-Mo-V high-speed tool steel, and (2) a vacuum-melted carburising grade, 'Modified M50', containing 3·25 per cent. of nickel; (3) 'L.P.D.' Cr-Mo-W-V vacuum- or air-melted hot-work die steel; (4) 'M1' Mo-Cr-W-V high-speed tool steel; (5) 'Matrix M2' Cr-Mo-W-V high-speed tool steel; (6) 'Nitralloy N' Ni-Cr nitriding steel; (7) 'Cobalt-Chrome' die steel; and (8) a titanium alloy. Steels (1), (4), (5) and (7) were through-hardened, steel (2) was carburised, steel (6) was nitrided, and the titanium

* *We shall be pleased to supply a free copy of this publication.*

alloy was carbide - flame - plated. The 'L.P.D.' specimens were either carburised or nitrided.

The extensive data presented relate to the following tests: hardness, impact and tensile tests at room temperature and at 500°, 600° and 700°F. (260°, 315° and 370°C.); tests to determine dimensional stability at 500° and 700°F. (260° and 370°C.) and corrosion- and oxidation-resistance at room-temperature and 500° and 700°F.; pinion-tooth fatigue tests at 700°F. (370°C.); and gear-roll tests (designed to determine resistance to scoring) at temperatures up to 700°F. In the gear-roll and corrosion tests use was made of 'Versilube F50' (a silicone) and of 'MLO 7056A' (a mineral oil).

In general the case-hardened steels were found to exhibit higher resistance to gear-tooth fatigue than the through-hardened specimens. The best results were obtained with carburised 'Modified M50', while 'Matrix M2' was the most satisfactory of the through-hardened materials. Fatigue-resistance of vacuum-melted specimens was slightly higher than that of the air-melted.

Resistance to scoring was governed by the type of oil used: the best results with the silicone oil were obtained with nitrided 'L.P.D.', but the performance of the 'M1' material rose from second to first place when mineral oil was used. Except for this reversal the resistance of the materials in this respect could be correlated with hardness. Dimensional stability is considered to offer no problem in designing gears in any of the materials tested, and the mild corrosion which occurred using 'Versilube F50' does not preclude the use of such oil. The oxidation that occurred was negligible.

The general conclusions are that, where scoring is the most important factor in designing high-speed gears, use should be made of nitrided 'L.P.D.' (in conjunction with the silicone) and 'M1' steel (with mineral oils). For low-speed heavily-loaded gears the carburised 'Modified M50' should be employed, irrespective of the oil. In applications requiring both scoring- and fatigue-resistance nitrided 'L.P.D.' is probably the best compromise.

Development of Nickel-containing Filler Wires for Welding High-Strength Steels

H. W. MISHLER and R. P. SOPHER: 'Development of High-Strength Filler Wires for Welding SAE 4130, 4140 and 4340 Steels.'

Wright Air Development Center, Tech. Report 56-550, Apr. 1957; 69 pp.

The increased use of high-strength low-alloy steels for aircraft components has necessitated the development of coated electrodes and filler wires capable of producing welds which will satisfy service requirements. Use of the inert-gas tungsten-arc and inert-gas consumable-electrode processes has, however, been hampered by the inadequacy of the filler wires available: porosity, weld-metal cracking and insufficient response to heat-treatment are among the difficulties encountered. The comprehensive research programme described in this report was initiated in an attempt to develop filler wires which would provide an effective solution to such difficulties, when used to weld the A.I.S.I. 4130, 4140 and 4340 grades of high-strength steel. The limits of composition for the three grades are shown in the table below.

The investigation was carried out in two phases. In the first phase an evaluation was made of commercially available filler wires, using the following criteria: resistance to weld-metal porosity and to weld-metal cracking; response of the weld metal to the standard heat-treatments applied to the basis metal; strength of the heat-treated joint; and toughness of the weld-metal.

The results of these tests and of analyses of the filler wires and weld-metal deposits (the latter enabling determination of the efficiency of alloy transfer across the arc, and hence providing the information necessary for modifications in composition which would compensate for loss of alloying elements during welding) were used in the second phase of the investigation as the basis for preparation of heats of steel from which experimental filler wires were drawn. Weld deposits made from these and commercial filler wires were then subjected to a series of tests, which included radiographic, hot-cracking, heat-treatment and (in the case of the most promising weld metals) transverse - tension, Charpy V - notch impact, and Jominy-hardenability tests. A few fatigue tests were also carried out. The main aim of the development and evaluation phases of the programme was to obtain two filler wires for each of the three steels, one of similar composition to that of the basis metal, the other of dissimilar composition. Full details are given of the seventeen filler wires studied and the test procedures used. The data obtained are presented *in extenso*.

Of the nine filler wires evaluated for use with A.I.S.I. 4130 steel up to $\frac{1}{4}$ in. (0·6 cm.) thick, only an experimental manganese-nickel-chromium-molybdenum filler wire* and one of a composition similar

* Carbon 0·18, silicon 0·44, sulphur 0·012, phosphorus 0·007, manganese 1·49, nickel 1·2, chromium 0·65, molybdenum 0·34, per cent.

Grade	C %	Mn %	Ni %	Cr %	Mo %
4130	0·28-0·33	0·4 -0·6	—	0·8-1·1	0·15-0·25
4140	0·38-0·43	0·75-1·0	—	0·8-1·1	0·15-0·25
4340	0·38-0·43	0·6 -0·8	1·65-2·0	0·7-0·9	0·2 -0·3

to that of the steel gave satisfactory welds. With ½-in. thick A.I.S.I. 4140 steel, best results were obtained using either a similar material or the manganese – nickel – chromium – molybdenum experimental steel as filler metal, while A.I.S.I. 4340 or experimental manganese-nickel-chromium-molybdenum-vanadium* filler wires proved satisfactory for welding 1-in. (2·5-cm.) thick A.I.S.I. 4340 steel.

HEAT- AND CORROSION-RESISTING MATERIALS

Recent Developments in Materials for High-Temperature Service

J. J. HARWOOD: 'New Developments in Metals and Ceramics for High-Temperature Service (above 1000°F.).'

Amer. Soc. Mechanical Engineers, Preprint 59-*MD*-2, 1959; 12 pp.

In this review the author briefly discusses current approaches to high-temperature problems, outlines the types of material at present available for high-temperature applications, and then attempts a comparative evaluation of pertinent classes of materials in relation to their capabilities, limitations, and potentialities for high-temperature aeronautical service.

The review, which is supplemented by data indicating the properties representative of the groups of material discussed, is in four sections, three of which cover materials for use in the following temperature ranges:

(1) 1000°–1500°F. (540°–815°C.)

The notes relate to titanium alloys, high-strength steels (low-alloy and hot-work die steels, martensitic and age-hardenable semi-austenitic stainless steels) and beryllium.

(2) 1200°–1800°F. (650°–980°C.)

Nickel-, cobalt-, and iron-base high-temperature alloys are briefly considered.

(3) Above 2000°F. (1095°C.)

A survey is given of the potentialities of refractory metals, various molybdenum-base and niobium-base alloys, cermets and ceramics. Use of refractory coatings to confer oxidation-resistance is discussed.

The last section of the paper is concerned with thermal protection systems, i.e., systems designed to cool a load-bearing structure to relatively low temperatures

Vacuum-Melted Austenitic Steel

SOC. AUTOMOTIVE ENGINEERS: 'Steel, Corrosion- and Heat-Resistant ; Consumable-Electrode Vacuum-Melted.'

A.M.S. Specification 5741, June 15, 1959.

The material covered is intended primarily for parts such as turbine discs and rotors, shafts, blades, vanes, bolts, flanges and other fittings which require high strength up to 1350°F. (732°C.) and oxidation-resistance up to 1500°F. (815°C.).

The composition is as follows: carbon 0·08 max., silicon 0·10–0·80, manganese 1·25–2·00, sulphur 0·030 max., phosphorus 0·040 max., chromium 12·00–15·00, nickel 24·00–28·00, molybdenum 1·25–2·25, titanium 2·70–3·30, boron 0·025–0·12, aluminium 0·25 max., copper 0·25 max., per cent.

The schedule gives details of heat-treatment (solution-annealing and precipitation-hardening) and lays down requirements for tensile properties, hardness and stress-rupture testing of the heat-treated material.

* Carbon 0·25, silicon 0·65, sulphur 0·012, phosphorus 0·008, manganese 1·17, nickel 1·83, chromium 1·17, molybdenum 0·8, vanadium 0·21, per cent.

Precipitation-Hardening Stainless Steel

SOC. AUTOMOTIVE ENGINEERS: 'Steel, Corrosion- and Moderate Heat-Resistant.'

A.M.S. Specification 5520, June 15, 1959.

The schedule covers sheet, strip and plate for use in parts in which corrosion-resistance, high strength and oxidation-resistance are required at temperatures up to 800°F. (427°C.), including parts which may require welding.

The compositional limits, per cent., are as follows: carbon 0·09 max., silicon 1·00 max., manganese 1·00 max., sulphur 0·03 max., phosphorus 0·04 max., chromium 14·00–16·00, nickel 6·50–7·75, molybdenum 2·00–3·00, aluminium 0·75–1·50.

Tensile properties, varying according to the thickness of the materials, and hardness values, are laid down for material in three conditions: (1) after annealing and air-cooling, (2) after transformation and precipitation hardening, and (3) after austenite-conditioning, sub-zero transformation, and precipitation-hardening.

Nickel-Chromium-base High-Temperature Alloy

SOC. AUTOMOTIVE ENGINEERS: 'Alloy, Corrosion- and Heat-Resistant; Consumable-Electrode or Vacuum-Induction Melted.'

A.M.S. Specification 5660, June 15, 1959.

Bars, forgings and forging stock for parts such as turbine rotors, shafts, blades, etc., requiring high strengths up to 1400°F. (760°C.) and oxidation-resistance up to 1600°F. (870°C.) are made from an alloy of the following composition: carbon 0·10 max., silicon 0·60 max., manganese 2·00 max., sulphur 0·030 max., phosphorus 0·030 max., chromium 11·00–14·00, nickel+cobalt 40·00–45·00 (cobalt, if determined, 1·00 max.), molybdenum 5·00–7·00, titanium 2·35–3·00, aluminium 0·35 max., boron 0·010–0·020, copper 0·50 max., per cent., iron remainder.

Solution-stabilisation and precipitation-hardening-treatments are specified, and tensile, hardness and stress-rupture requirements are laid down.

Solid-Solution, Carbide and Precipitation Hardening of Nickel- and Cobalt-base Alloys

F. EBERLE, J. H. HOKE, E. J. ROZIC and W. E. LEYDA: 'The Strengthening of Austenitic Solid Solutions.'

Wright Air Development Center, Tech. Report 58-28, Apr. 1958; 60 pp.

High-temperature alloys are, in general, strengthened

by one or more of the following methods: solid-solution hardening (introduction of 'incongruous' atoms into the lattice); carbide hardening (*via* additions of carbon and strong carbide-forming elements which, after suitable heat-treatment, precipitate in the matrix as complex carbides); and precipitation hardening (*via* alloying additions which precipitate as intermetallic compounds). The literature, however, contains relatively little data either on the creep-rupture strengths of the matrix or basis compositions or on the relative effectiveness of the three types of strengthening mechanism. This report presents the results of a systematic investigation of these factors, carried out with a view to development of stronger and more economical alloys suitable for applications at temperatures of 1600° and 1800°F. (870° and 980°C.).

The study was conducted in two stages. In the first, vacuum-melted carbon-free solid solutions of Fe-Cr-Ni, Cr-Ni, Fe-Cr-Co, Cr-Co and Fe-Cr-Ni-Co base were tested, over their entire range of alloying combinations at chromium levels of 20, 30 and (with the exception of the Fe-Cr-Ni-Co alloys) 15 per cent., to determine room-temperature mechanical properties and creep-rupture strength at 1600° and 1800°F. (870° and 980°C.). The optimum basis compositions revealed by these screening tests were then, in the second stage of the study, subjected to solid-solution-hardening and precipitation-hardening treatments, as a means of assessing the relative potency of these treatments and of indicating the way to development of alloys having improved properties at 1800°F. (980°C.). All the alloys studied were subjected to a solution heat-treatment at 2000°F. (1095°C.) before testing, a procedure which, the authors note, would not produce optimum properties in the precipitation-hardened alloys. Moreover the funds and time available permitted exploration of the effects of strengthening additions at only two concentrations.

On the basis of the preliminary screening tests, two compositions were selected for consideration in the second phase of the study: 10Fe-20Cr-70Ni and 20Cr-80Co. The two alloys exhibited an interpolated 100-hour rupture-strength at 1800°F. (980°C.) of, respectively, 2000 and 2350 p.s.i. In the second phase of the investigation stress-rupture tests were carried out only at 1800°F. (980°C.). A brief indication of the scope of the latter programme is given below.

Effects of Solution-Hardening

with alloying additions (3 and 6 per cent.) of manganese, silicon, cobalt, molybdenum, tungsten, niobium and tantalum.

In the case of the nickel-base composition the only elements to produce a slight improvement in rupture-strength were the austenite stabilisers, manganese and cobalt; the elements with ferrite-forming tendencies even caused a decrease in rupture-strength.

The rupture-strength of the cobalt-base alloy was improved by all the additions, introduction of silicon and tantalum producing a substantial improvement.

Effect of Carbon Additions

Additions of up to 0·75 per cent. of carbon halved the rupture strength of the Fe-Cr-Ni alloy, but slightly increased that of the cobalt-base composition.

Complex Carbide Hardening

was successfully applied to both basis compositions, but appeared to offer more promise in relation to the cobalt-base alloy. A maximum increase of 50 per cent. was obtained in the rupture-strength of the nickel-base alloy by addition of 0·25 per cent. of carbon and 3 per cent. of titanium; in the cobalt-base alloy an addition of 0·25 per cent. of carbon and 6 per cent. of molybdenum improved rupture-strength by 125 per cent.

Precipitation Hardening with Additions forming Intermetallic Compounds

The strengthening effects of titanium and/or aluminium were studied. An aluminium addition of 3 per cent. slightly weakened both alloys. The other results indicate that the cobalt alloy is strengthened only by titanium, and that the strength of the nickel alloy is improved by Ti+Al additions.

Effect of Carbon Additions on Precipitation-Hardened Alloys

Carbon additions considerably strengthened the compound-hardened nickel-base alloy, but reduced the rupture strength of the cobalt-base composition. The effects of carbon+(Ti+Al) additions on the properties of nickel alloys are considered worthy of further investigation. A nickel-base alloy of the following composition exhibited a 100-hour rupture-strength at 1800°F. (980°C.) of 6800 p.s.i., the highest of all the alloys tested: nickel 72·8, chromium 19, titanium 4, cobalt 1·5, aluminium 1·16, carbon 0·29, per cent.

Effect of Combined Complex-Carbide and Intermetallic-Compound Hardening

Addition of carbide formers lowered the rupture-strength of a carbon-containing precipitation-hardened nickel-base alloy (see composition above) and also, though to a lesser extent, that of a similarly hardened cobalt alloy.

Oxidation tests on representative nickel- and cobalt-base alloys indicated that the former materials exhibited somewhat higher oxidation-resistance.

The data obtained from the test programme are tabulated and evaluated in detail. The authors' general conclusions are that alloys of Fe-Cr-Ni or Cr-Ni base offer most promise of development for use at 1800°F. (980°C.), and, in comparison with the other base compositions studied, offer the following advantages:

(1) Superior cold ductility.

(2) Superior oxidation-resistance at lower chromium contents.

(3) Ability to develop a higher strength level.

Influence of Static Pre-Straining on the Fatigue Properties of Nickel-containing Steels at High Temperatures

F. H. VITOVEC: 'Effect of Static Prestrain on the Prot-Fatigue Properties of Unnotched Materials at Room and Elevated Temperature.'

Wright Air Development Center, Tech. Report 58-214, July 1958; 60 pp.

Earlier studies of the effect of pre-straining on fatigue characteristics have been concerned mainly with determination of the influence of cold-work and residual stress on properties at room temperature. At elevated temperatures the duration for which a material is subjected to static loading is of particular importance, and short-time pre-strain may influence fatigue properties differently from long-time pre-strain due to creep. The study now reported was carried out to establish the effects of short-time and creep pre-strain on the fatigue properties of aircraft structural materials at elevated temperatures.

Four materials were investigated: two aluminium alloys, the chromium-nickel-molybdenum heat-resisting steel '16-25-6' and the cobalt-nickel-chromium - base high - temperature alloy 'S - 816'. Specimens from the three groups were tested in, respectively, the extruded, the 'hot-cold-worked' and the aged conditions. Tests were made on both notched and unnotched specimens of the '16-25-6' steel and the aluminium alloys. Pre-straining and fatigue testing were carried out at the same temperatures. The aluminium specimens were subjected to short-time pre-straining at 75° or 300°F. (24° or 150°C.) and to creep at 300° or 500°F. (150° or 260°C.). 'S-816' was pre-strained at 1500°F. (850°C.) and '16-25-6' at 1200°F. (650°C.). In short-time straining, loads were applied to achieve strains of up to 10 per cent. in five to ten minutes. The stresses used for creep pre-straining corresponded to those producing rupture in 100-200 hours at the test temperature. Fatigue properties were determined immediately after pre-straining, using a Prot-type testing machine with a single loading rate of 0·01 p.s.i. per cycle.

The author's survey of the data obtained from these tests leads him to the general conclusion that the main factors influencing the Prot failure stress were residual stresses, metallurgical reactions, and cracks produced by pre-straining. Strain-hardening and relaxation of residual stress appear to be of secondary importance under the test conditions studied. The particular conclusions drawn with respect to the nickel-containing materials are indicated below.

The fatigue-strength of the '16-25-6' steel decreased significantly at first with increase in short-time pre-strain. A minimum occurred at about 2·7 per cent. pre-strain, after which the fatigue-strength increased to the original values, a trend indicating reversal of precipitation and renewed precipitation. The fatigue-strength of the 'S-816' alloy was increased by short-time pre-straining. Prior creep resulted in a slight decrease in the fatigue-strength of the '16-25-6' steel, and the fatigue-strength of the 'S-816' alloy was at first slightly increased and then decreased by prior creep. Short-time tensile pre-straining of notched specimens increased the fatigue-strength of all the materials tested; compressive pre-straining had the opposite effect. Creep tensile pre-straining of notched specimens of '16-25-6' steel resulted in a significant increase in fatigue-strength, indicating that a relatively slow relaxation of residual stresses occurred at the test temperature.

Influence of Minor Additions of Other Elements on a Nickel-Chromium Alloy

N. T. GUDTSOV, R. I. TRUBETSKOVA and M. L. BERNSHTEIN: 'The Effect of Small Additions of Boron, Calcium, Niobium, Zirconium and Cerium on the Structure and Properties of High-Nickel Creep-Resistant Alloy.'

Proizvodstvo i Obrabotka Stali i Splavov, Moskov. Inst. Stali im. I.V. Stalina, Sbornik, 1958, vol. 38, pp. 495-516.

The amounts of the respective alloy elements introduced into the parent material were, respectively, boron 0·005, calcium 0·1, niobium 0·5, zirconium 0·2, cerium 0·01, per cent. The influence of these additions was studied in relation to the structure of the alloy in various conditions of heat-treatment.

For details see *Chemical Abstracts*, 1959, vol. 53, May 10, p. 7927.

The exact composition of the parent alloy (type 36KhTYu) is not given in the abstract.

Influence of the Degree of Stabilisation on Precipitation Reactions in Niobium-containing Austenitic Steels

K. BUNGARDT, G. LENNARTZ and K. WETZLAR: 'Influence of the Degree of Stabilisation on Precipitation Reactions in Niobium-containing Austenitic Chromium-Nickel and Chromium-Nickel-Molybdenum Steels.'

Archiv f. d. Eisenhüttenwesen, 1959, vol. 30, July, pp. 429-34.

In recent years a series of investigations have been in progress to determine the influence, on the creep behaviour of ferritic and austenitic steels, of the progress of precipitation, and the changes in the nature of the precipitates, during creep testing. (See, for example, papers by the present authors referred to in *Nickel Bulletin*, 1958, vol. 31, No. 9-10, p. 271; *ibid.*, 1959, vol. 32, No. 4, pp. 119-120.)

Comprehensive studies of titanium- or niobium-stabilised austenitic steels were, in particular, conducted by KOCH *et al* (see abstract in *Nickel Bulletin*, 1959, vol. 32, No. 2, p. 49), and the influence, in this respect, of the degree of stabilisation, and of the nickel and molybdenum contents, was reported *in extenso* by BUNGARDT and SYCHROVSKY (*Stahl u. Eisen*, 1955, vol. 75, Jan. 30, pp. 25-39; *Nickel*

Bulletin, 1955, vol. 28, No. 4, pp. 68–9). The work now recorded was initiated in an attempt to throw more light on the progress of precipitation in relation to the degree of stabilisation and to the nickel, molybdenum, silicon and manganese contents of the steel.

The composition of the ten steels investigated (six of which were of 18–8–Mo type) varied within the following range: carbon 0·004–0·108, silicon 0·14–0·56, manganese 0·09 – 1·42, chromium 14·98 – 16·77, nickel 16·1–18·56, molybdenum <0·1–2·18, niobium 0·39–0·97, nitrogen 0·008–0·028, per cent. The degree to which the steel was over- or under-stabilised was assessed by relating the niobium content to the sum total of the carbon and nitrogen contents of the steel. Specimens of each steel were solution-annealed at 1100°C., water-quenched, and then soaked at 700°C. for from 100 to 5,000 hours. The main creep tests, which were carried out at 700°C., were supplemented by some at 650°C. Determination and structural identification of the precipitates present, and study of microstructural changes, were effected by X-ray and chemical examination of electrolytically isolated precipitates.

The results of the investigation, which are presented in detail in the paper, show that variations in the degree of stabilisation have a pronounced effect on precipitation in both molybdenum-containing and molybdenum-free chromium-nickel steels: even a slight degree of under-stabilisation led in both cases to precipitation of stable chromium carbides.

Since the solubility of the niobium at 700°C. is less than 0·02 per cent., even a slight degree of over-stabilisation was sufficient, in the case of the molybdenum-free steels, to cause precipitation of iron niobide. Increasing the nickel content of these steels from 13 to 16 per cent. suppressed the occurrence of sigma phase, even in creep tests up to 10,000 hours at 700°C.

Irrespective of the degree of stabilisation, a Laves phase of $MgZn_2$ type precipitated in the 16–16 molybdenum-containing steels. In the under-stabilised steels this phase consisted of Fe_2Mo, but when the steel was over-stabilised the phase took the additional niobium into solution and the size of the lattice parameter increased. Sigma phase was also precipitated in the over-stabilised molybdenum-containing steels, the formation of which was accelerated and intensified by increase in the silicon and manganese contents. On the other hand, an increase in the nickel content to higher than 18 per cent. prevented the phase's formation.

The solubility of niobium carbide in the molybdenum-containing solid solution is higher at 1100°C. than in the molybdenum-free steels, a factor which led to a more pronounced formation of metastable chromium carbides in the over-stabilised molybdenum-containing steels than in the corresponding molybdenum-free grades.

Stressing during creep testing had the effect of accelerating precipitation without influencing the precipitation reaction itself.

Mechanical Properties of Structural Materials after Rapid Heating

H. E. DEDMAN, E. J. WHEELAHAN and J. R. KATTUS: 'Tensile Properties of Aircraft-Structural Metals at Various Rates of Loading After Rapid Heating.' *Wright Air Development Centre, Tech. Report* 58–440, *Part I*, Nov. 1958; 197 pp.

The work reported represents one of a series of investigations which are being carried out to evaluate the mechanical properties of aircraft structural materials under conditions of rapid heating, short times at temperature and moderate to high rates of loading (i.e., under conditions to which the materials are often subjected in aeronautical applications).

Seventeen of the materials selected for investigation had been previously tested under similar conditions, and the results were reported in *W.A.D.C. Tech. Report* 55–199, *Parts I–III* (see abstract in *Nickel Bulletin*, 1958, vol. 31, No. 1, pp. 17–18). These materials included: aluminium alloys; magnesium alloys; titanium alloys; 18–8-type chromium-nickel steels (Types 301 and 321); '17–7 PH' stainless steel; low-alloy chromium-molybdenum steel; and carbon steel. In the previous investigation, however, stress/strain curves were determined only to about 1·5 per cent. strain; beyond that point the load alone was recorded. The work now described was initiated to obtain full stress/strain curves for all the materials under the test conditions previously employed. The main emphasis was on accurate determination of 0·2 per cent. offset yield strength and ultimate tensile strength. Modulus of elasticity, percentage elongation, proportional limit and full stress/strain curves were determined with sufficient accuracy to establish trends.

In the present investigation five additional sheet materials, all heat-treated to high strength levels, were also fully evaluated: the chromium-molybdenum-tungsten tool steel 'Chro-Mow', the chromium-nickel-molybdenum-vanadium tool steel 'Thermold J', the chromium-molybdenum-vanadium tool steel 'Peerless 56', the chromium-nickel-molybdenum stainless steel 'AM-350', and a titanium alloy. The test specimens were heated to test temperature within 10 seconds, held at temperature for times ranging from 10 to 1800 seconds, and loaded at strain rates from 0·00005 to 1·0 in./in./sec. In general, test temperatures ranged from room-temperature to 600°F. (315°C.) in the case of the aluminium and magnesium alloys, to 1000°F. (540°C.) in the case of the titanium alloys, and to 1200°F. (650°C.) in the case of the steels and cobalt- or nickel-base alloys.

The data obtained are presented *in extenso*, but the discussion of the results and the authors' conclusions relate only to the five materials which were not covered by the previous test programme. A detailed analysis of the short-time tensile properties of all the materials evaluated will be published in Part II of the report.

The results show that, at the lower test temperatures, the strength properties of the test alloys tended to

increase slightly, or to remain constant, with increase in rates of strain. Near the maximum test temperatures the effects of strain rate became much more pronounced, the strength properties increasing greatly with increase in strain rate. Probably as a result of structural changes associated with tempering and with over-ageing, the strength of the steels decreased with increase in holding times at 1200°F. (650°C.). The tensile properties exhibited by the steels at lower temperatures, and by the titanium alloy at all test temperatures, were not influenced by variations in time at temperature.

Compressive, Bearing and Shear Creep-Rupture Properties at Elevated Temperatures

L. A. YERKOVICH: 'Investigation of the Compressive, Bearing, and Shear Creep-Rupture Properties of Aircraft Structural Metals and Joints at Elevated Temperatures.'

Wright Air Development Center, Tech. Report 54-270, *Part V*, Dec. 1958; 84 pp.

The report is one of a series recording the results of work to determine the creep behaviour of aircraft structural materials under the influence of shear, bearing, and compressive stresses (see abstract of an earlier report in *Nickel Bulletin*, 1959, vol. 32, No. 5, p. 133). The main aim of the investigation was to obtain data which would permit correlation of tensile behaviour with behaviour under other loading conditions. These basic data would then be used to predict the behaviour of riveted joints during creep deformation in tension, bearing and shear.

Two materials were selected for study: the age-hardenable chromium-nickel-molybdenum stainless steel 'PH 15-7 Mo' (in the hardened condition), and a titanium alloy. Specimens of both materials were subjected, in sheet and bar form, and under tensile, bearing, and compressive loads, to short-time tests at room- and elevated temperatures, and to creep tests at 700°, 800° and 900°F. (370°, 425° and 480°C.). Creep-rupture tests were conducted also on double-type riveted joints designed to fail preferentially, either by bearing creep-rupture of the riveted sheet, or by shear creep-rupture of the rivet.

The large amount of data obtained from the investigation indicates that the creep properties of both materials are similar under bearing and shear stress to those which they exhibit during tensile testing: deformation occurs in three characteristic creep-rate stages. Good agreement was obtained between correlations of creep-rupture data and correlations of short-time strength when the short-time characteristics were determined under a controlled strain rate of 0·5 per cent. per minute. Within the rupture times of interest, and particularly at lower test temperatures, both materials exhibited creep-rupture stress sensitivity in bearing and shear as well as in tension, and at 700°F. (370°C.), for example, stress variations of a few per cent. had a considerable influence on time-to-rupture. At this lower test temperature, moreover, stresses considerably higher than the yield strengths had to be applied to produce rupture in less than 500 hours.

The results of the tests on riveted joints suggest that creep-rupture performance of joints cannot be predicted with any reasonable degree of accuracy when failure occurs by shear in the rivet. When joints were designed to rupture in bearing, on the other hand, it was found that creep-deformation characteristics, as well as time-to-rupture, were predictable from the basic bearing creep-rupture properties of the bearing element.

Tensile, Compressive, Bearing and Shear Properties of Sheet Steels at Elevated Temperatures

J. R. KATTUS, J. B. PRESTON and H. L. LESSLEY: 'Determination of Tensile, Compressive, Bearing and Shear Properties of Sheet Steels at Elevated Temperatures.'

Wright Air Development Center, Tech. Report 58-365, Nov. 1958; 274 pp.

The purpose of the investigation was to determine the effects of exposure times of up to 1000 hours at test temperature on the elevated-temperature tensile, compressive, bearing and shear properties of structural steels used for aeronautical applications.

Six steels were studied: the precipitation-hardenable chromium - nickel stainless steels 'A-286' and '17-7 PH'; the low-alloy chromium-nickel-molybdenum-vanadium steel 'Thermold J'; two chromium stainless steels (Types 420 and 422); and the low-alloy chromium - molybdenum - vanadium steel '17-22A(S)'. With the exception of the '17-7 PH' steel (which was evaluated in the RH 950 condition) all the materials were tested in the quenched-and-tempered condition. 'A-286' was tested at temperatures in the range 75°-1200°F. (24°-650°C.), 'Thermold J' in the range 75°-1100°F. (24°-595°C.), and the other steels in the range 75°-1000°F. (24°-540°C.).

The strength properties and moduli of elasticity of all the steels decreased with rise in temperature. Those of the 'Thermold J', Type 420, Type 422, and '17-22A(S)' specimens tended to decrease, by varying amounts, with increase in the time of exposure at the higher test temperatures, and these decreases are believed to be associated with structural changes resulting from tempering. At lower temperatures the properties of these materials did not vary significantly with exposure time, indicating that the structures were stable at these temperatures. The strength properties of the precipitation-hardenable steels 'A-286' and '17-7 PH' varied somewhat erratically with increase in exposure times at the higher test temperatures, due probably to ageing phenomena.

Comparison and correlation of the various properties studied showed that their ratio relationships for equivalent test conditions varied somewhat with differences in the material, temperature and time of exposure. It is not, therefore, considered possible to calculate, with any high degree of accuracy, design data for a given property by means of a

general factor relating that property to another property that had been determined experimentally. Precise and reliable data on the tensile, compressive, bearing and shear properties of aircraft-structural materials can at present be obtained only by testing under the specific conditions concerned.

Effect of Cold-Working on Nickel-Chromium Alloys

M. L. BERNSHTEIN and Z. V. SVISTUNOVA: 'The Effect of Cold-Working on the Structure and Properties of Nickel-Chromium Creep-Resistant Alloy of Type EI 437.'

Proizvodstvo i Obrabotka i Splavov, Moskov. Inst. Stali im. I.V. Stalina, Sbornik, 1958, vol. 38, pp. 517–35.

Study of the effect of cold-working, with cross-section reduction of 5, 25, 50 or 75 per cent., on transformations in, and properties of, a nickel-chromium alloy containing carbon 0·075, silicon 0·47, manganese 0·22, sulphur 0·0047, phosphorus 0·009, chromium 20·5, cerium 0·04, titanium 2·62, aluminium 0·56, copper 0·02, iron 0·01, per cent.

The specimens were annealed at 1080°C. for 8 hours, cooled in water or air, or furnace-cooled, and afterwards subjected to cold-working. Hot hardness at the ageing temperatures of 500°, 600°, 700° or 800°C. was measured after 5, 50, 500, 5,000 or 50,000 minutes, and measurements were made of electrical resistivity.

For general observations on the results of the measurements see *Chemical Abstracts*, 1959, vol. 53, May 10, p. 7934.

High-Temperature Properties of 'Hastelloy B' and 'Hastelloy W'

C. R. KENNEDY and D. A. DOUGLAS: 'High-Temperature Mechanical Properties of 'Hastelloy B' and 'Hastelloy W'.

U.S. Atomic Energy Commission, Report ORNL-2402, 1959; 81 pp.

Nickel-molybdenum-base alloys exhibit a combination of properties (high-resistance to corrosion in fused salts, excellent high-temperature strength, and relative freedom from objectionable high-cross-sectional elements) which render them of interest as possible materials of construction for a circulating-fuel reactor. The prerequisites for such applications are resistance to the high temperatures and corrosive environments involved, allied with sufficient ductility to withstand the thermal strains introduced into the structure by temperature and load fluctuations during operation of the reactor. Data available on the high-temperature properties of interest in this respect were, however, derived from conventional tests in air. The investigation now reported was carried out to evaluate the nickel-chromium-base alloys in the environment to which they would be exposed in service.

The compositions of the two alloys tested, 'Hastelloy B' and 'Hastelloy W', are given in the table below.

Creep specimens machined from 0·06-in. (1·5-mm.) sheet were solution-annealed in hydrogen for 2 hours at 2100°F. (1150°C.), and most of the specimens were tested in this condition. To determine, however, the effects of prior ageing on the creep properties of 'Hastelloy B', some of the test pieces were aged, before testing, for 100 hours at 1300°F. (705°C.) and 40 to 70 hours at 1500°F. (815°C.).

For design purposes, and as a basis for comparison of the effects of environment, the creep-rupture properties of the two alloys, in the solution-annealed condition, were determined in argon. Creep tests were conducted at temperatures in the range 1300°–1800°F. (705°–980°C.), and, since the compositions and stress-rupture properties of the two alloys are very similar, the majority of the tests to determine the effects of environment were carried out on 'Hastelloy B'. The environments studied in this connexion were: argon, sodium, air, hydrogen, and two fused salts ($NaF/ZrF_4/UF_4$, $NaF/KF/LiF/UF_4$). All the creep specimens were subjected to metallographic examination, and hardness/time-at-temperature and ductility/time-at-temperature curves were also determined.

The two alloys were found to have excellent corrosion-resistance to the fused salts and satisfactory high-temperature strength. Phase transformations occurring within the operating temperature range of the reactor caused severe loss of ductility, however, and on that account the materials are considered unsafe for reactor construction.

The following general conclusions are drawn from the results obtained:

'(1) Both alloys possess reasonably good high-temperature strength properties.

'(2) 'Hastelloy B' and 'Hastelloy W' exhibit a severe loss of hot ductility at temperatures where the beta transformation occurs.

'(3) The change in composition found in 'Hastelloy W' results in a suppression of the beta and gamma transformation.

'(4) Room-temperature tests indicate that both alloys fail to soften after 4,000 hours at 1300°F. (705°C.) and 1500°F. (815°C.).

'(5) Room-temperature ductility is apparently affected by the formation of unidentified phases of

Alloy	Mo %	Fe %	Mn %	Si %	Co %	V %	Cr %	C %	P %	S %	Ni %
'Hastelloy B'	27·5	4·85	0·87	0·65	0·34	0·32	0·3	0·05	0·003	—	rem.
'Hastelloy W'	24·3	5·25	0·39	0·2	0·82	0·27	4·71	0·03	0·008	0·02	rem.

molybdenum and minor alloying constituents such as iron.

'(6) 'Hastelloy B' and 'Hastelloy W' are very resistant to corrosion by both fused salts.

'(7) Air and the $NaF/ZrF_4/UF_4$ fused salt are two environments which strengthen 'Hastelloy B' and 'Hastelloy W' by surface alteration when there is a large surface/volume-of-metal ratio.'

Properties of 'Hastelloy B': Influence of Thermal Cycles Involved in Welding

E. G. THOMPSON: 'The Effect of Simulated Weld Thermal Cycles on the Properties of 'Hastelloy B'. *U.S. Atomic Energy Commission, Report ORNL-2582*, Oct. 28, 1958; 65 pp.

Much research has been carried out to determine the causes of cracking in the heat-affected zones of weldments produced in stainless steel and nickel- or cobalt-base alloys. Since the fractures are intergranular in nature, cracking has been attributed to the presence of brittle or low-melting grain-boundary films, but the results of previous investigations have proved inconclusive and conflicting. It was therefore decided to study the influence, on the properties and microstructure of the heat-affected zones of such materials, of exposure to a rapid thermal cycle characteristic of that involved in welding. The results of tests on the nickel-molybdenum-base alloy 'Hastelloy B' are recorded in the present report. (For the salient findings of a germane investigation on 'Inconel' and 'Inconel X' see abstract in *Nickel Bulletin*, 1959, vol. 32, No. 6, p. 190.)

To determine their hot-ductility at temperatures representing the peak of the thermal cycle at various distances from the fusion zone of the weld, specimens of the alloy were fractured, using a time-temperature controller and hydraulic testing device which are described, at 1500°, 1800°, 2000°, 2200°, 2250°, 2300°, 2350° and 2400°F. (815°, 980°, 1095°, 1205°, 1230°, 1260°, 1285° and 1315°C.). Other specimens were heated rapidly to peak temperatures of 2300°, 2350° and 2400°F. (1260°, 1285° and 1315°C.) and then ruptured, during the cooling portion of the thermal cycle, at 1500°, 1800°, 2000°, 2220° and 2300°F. (815°, 980°, 1095°, 1205° and 1260°C.). The data determined for reduction-in-area, load and strain were correlated with microstructural changes occurring as a result of the thermal cycle involved.

The structure of the alloy in the as-received condition was found to consist of a nickel-rich solid solution of molybdenum (α) and a single secondary phase concluded to be an intermetallic compound NiMo (δ).

The ductility of the specimens tested was unimpaired by thermal cycles involving a maximum temperature of up to 2250°F. (1230°C.). At 2300°F. (1260°C.), however, ductility began to decrease, and at 2350°F. (1285°C.) and 2400°F. (1315°C.) the material was completely brittle. Thermal cycling to a peak temperature of 2300°F. (1260°C.) caused no loss in ductility of 'Hastelloy B' at lower temperatures, but cycling to 2350°F. (1285°C.) adversely affected hot-ductility in the region of 2300°F. (1260°C.), and exposure at a peak temperature of 2400°F. (1315°C.) was found to be slightly deleterious to ductility at temperatures in the range 1800°-2000°F. (980°-1095°C.) and to cause severe embrittlement at higher temperatures.

Tensile strength was influenced only by the 2400°F. (1315°C.) thermal cycle, which caused about a 10 per cent. loss in strength at all the test temperatures. Strain measurements were found to be inadequate as a means of determining the effect of the thermal cycles.

The loss in ductility exhibited by 'Hastelloy B' after cycling to 2350°F. (1285°C.) is attributed to the formation of grain-boundary films. The severe loss in ductility and mild decrease in tensile strength resulting from exposure at 2400°F. (1315°C.) were, it is considered, caused by formation of a eutectic along the grain boundaries. It is concluded that this eutectic formed at a minimum temperature of 2350°F. (1285°C.), and, as indicated by its effect on the ductility of the alloy, must be weaker than the higher-melting matrix at temperatures above 2000°F. (1095°C.).

Heat-Resistance of Welded Joints in a Nickel-Chromium-base Alloy

B. I. MEDOVAR, A. N. SAFONNIKOV and R. O. LENTS: 'High-Temperature Strength of Welded Joints in the Nickel-Chromium Alloy Kh2ON8OT3Yu (EI-437B).'

Automaticheskaya Svarka, 1959, Feb.
Translated in *Automatic Welding*, 1959, Feb., pp. 1-20.

References in the literature indicate that submerged-arc welding with fused silicate fluxes is unsuitable for use with nickel-chromium-base heat-resisting alloys (e.g., of the 'Nimonic' series), due to the highly oxidising nature of the flux and its adverse effects on the aluminium, titanium, and chromium contents, and hence on high-temperature properties. In the present paper data are tabulated illustrating the high-temperature properties obtained in joints produced, using oxygen-free fluoride fluxes, by submerged-arc and electro-slag welding of a nickel-chromium heat-resisting alloy.

The composition limits of the alloy, designated 'EI-437B', are given as: chromium 19-22, iron 1 max., titanium 2·3-2·7, aluminium 0·55-0·95, boron 0·01, carbon 0·06 max., silicon 0·65 max., manganese 0·35 max., sulphur 0·007 max., phosphorus 0·015 max., per cent., remainder nickel. The fluxes were of CaF_2/NaF or CaF_2/Al_2O_3 type ('ANF-5' and 'ANF-6'), and the tests were conducted on joints which were 12-20 mm. thick (produced by submerged-arc welding) or (using electro-slag welding) 100 mm. thick. The data tabulated were derived from tests designed to evaluate the short- and long-time high-temperature mechanical properties of the joint in the age-hardened condition, and to determine the influence, on long-time strength,

of cold-working and of duration of heat-treatment.

The following conclusions are drawn:

'(1) The automatic arc welding of heat-resistant nickel-chromium alloys enables welded joints to be obtained with high heat-resistance, due to the use of oxygen-free fluoride fluxes.

'(2) When welding 10–20 mm.-thick 'EI-437B' alloy by submerged arc, using EI-437A electrode wire (a boron-free alloy, which otherwise is of similar composition to the basis metal), the long-time strengths of the welded joints are not less than 80–85 per cent. of those of the parent metals.

'(3) The electro-slag welding of heat-resistant nickel-chromium alloys, using plate-type electrodes, enables welded joints with high heat-resistances to be produced when fluoride-base non-oxidising fluxes are used, the weld strength being about 0·9 times that of the parent metal.

'(4) There are grounds for the assumption that welded joints in 'EI 437B' alloy up to 20 mm. thick can also be given high heat-resistance if the duration of heating, and also possibly the duration of the ageing process, are reduced to some extent.

'(5) The preliminary cold-working of welded joints before hardening results in some increase in heat resistance.'

Protection of Molybdenum from Oxidation at High Temperatures

'Coated Molybdenum Withstands 1800°F.'

Materials in Design Engineering, 1959, vol. 50, July, pp. 154, 156, 158.

In this brief *résumé* of a paper by M. A. LEVINSTEIN (presented at a recent meeting of the American Society of Metals) reference is made to work carried out by the General Electric Company, Ohio, on the development of oxidation-resistant coatings for molybdenum turbine blades.

Of the fifty coatings evaluated, one incorporating the following four layers gave the most promising results: (1) a chromium electrodeposit intended to provide a diffusion-type bond, (2) a nickel electrodeposit introduced to improve ductility, (3) a fused nickel-silicon-boron coating (as a bonding layer for the subsequent 'Nichrome' cladding), and (4) a 0·006-in. (0·15-mm.) brazed layer of 'Nichrome' hard-surfaced with a chromium-tungsten-cobalt-boron alloy.

In the coating process the turbine blade is first electropolished in sulphuric acid and then chromium plated. The chromium deposit is vapour-blasted to remove surface oxides and nickel plated, after which the blade is heat-treated in dry hydrogen at 2100°F. (1150°C.), as a means of diffusing the chromium to the molybdenum and the nickel to the chromium. The nickel coating is grit-blasted and flame-sprayed with the nickel-silicon-boron alloy. The flame-sprayed coating is then fused to the nickel deposit in a special retort, and finally the 'Nichrome' layer is brazed to the fused nickel-silicon-boron coating.

Blades coated by this technique were subjected to engine tests, under steady-state conditions at 1800°F. (980°C.) and under cyclic conditions at 1900°F. and 2000°F. (1035° and 1095°C.). Of the ninety-six blades tested for fifty hours under steady-state conditions, two failed as a result of impact damage to the coating. In the cyclic tests the chief cause of failure was thermal fatigue.

Thermal-Fatigue Cracking of Gas-Turbine Blading

D. F. SPRINGSTEEN, C. A. GYORGAK and J. R. JOHNSTON: 'Origin and Development of Leading-Edge Cracks in Turbojet Engine Buckets.'

Nat. Advisory Committee for Aeronautics, Research Memorandum E57C12, May 20, 1957; 37 pp.

This is an earlier report in a series of investigations to which reference was made in *N.A.S.A. Memorandum* 4-7-59*E*; see *Nickel Bulletin*, 1959, vol. 32, No. 8, p. 257. The nature of cracks observed in the leading edge of gas-turbine blading is discussed, and probable contributory causes are reviewed.

The tests reported were designed to study the origin and mode of development of the cracks. Particular attention was directed to determining the time and location of the crack initiation, the length of time between onset of cracking and fracture of the blade, the variation in crack-resistance of various high-temperature materials, and the effect of certain special treatments on crack-resistance.

Blading of six materials was tested: 'S-816', 'M-252', 'Inconel 550', 'Inconel 700', 'Hastelloy R-235' and 'Jetalloy 1570'. Special treatments applied included electropolishing, annealing, and re-heat-treating to reduce residual stresses introduced by fabrication. Some blades of 'S-816' alloy were aluminised.

Details are given of treatment and of test conditions. The latter involved simultaneous exposure of all the blades to cycles of 15 minutes at rated speed and approximately 5 minutes at idle speed, in a J 47-25 engine. Inspection methods are also fully described.

The results indicated that blading of all the materials tested was subject to leading-edge cracking. Total operating time after onset of cracking varied considerably (55 hours to 245 hours). The special treatments applied appeared to be ineffective in improving either crack- or fracture-resistance.

Analysis of factors potentially influencing development of the cracking phenomenon indicated that the probable primary cause was thermal fatigue. (It will be noted that the later investigation, to which reference was made in the August issue of the *Nickel Bulletin*, confirmed this conclusion.)

Tubing for High-Temperature Fluid-Transport Service

SOC. AUTOMOTIVE ENGINEERS: 'Alloy Tubing, Seamless, Corrosion- and Heat-Resistant.'

A.M.S. Specification 5582, June 15, 1959.

The material covered by the specification is for

use in fluid lines requiring high strength up to 1500°F. (815°C.) and oxidation-resistance up to 1800°F. (980°C.).

Compositional requirements: nickel+cobalt 70·00 min. (cobalt, if determined, 1·00 max.), chromium 14·00–17·00, carbon 0·08 max., silicon 0·50 max., manganese 1·00 max., sulphur 0·010 max., niobium +tantalum 0·70–1·20, titanium 2·25–2·75, aluminium 0·40–1·00, iron 5·00–9·00, copper 0·50 max., per cent. Properties are specified for the material in the cold-drawn and annealed condition, and flarability and pressure tests are specified. Tensile properties are specified for precipitation-hardened tubing and provision is made for a stress-rupture test at 1350°F. (732°C.).

Creep Testing of Steels Used in Steam Plant

W. RUTTMANN and R. SCHINN: 'Summary of the Results of Co-operative Work on 'Long-Time Creep Tests': Conclusions with Respect to Steam-Plant Construction.'

Mitteilungen der Vereinigung der Grosskesselbesitzer, 1958, No. 57, Dec., pp. 411–20.

The work with which the paper is concerned was begun in 1950, under the aegis of the Verein Deutscher Eisenhüttenleute, the Wasserrohrkessel-Verband, the Fachgemeinschaft Kraftmaschinen and the Vereinigung der Grosskesselbesitzer. Many of the specimens have been under test for more than 60,000 hours, and the present programme covers more than 1,000 tests at temperatures in the range 450°–750°C.

The data on which the authors conclusions are based were derived from tests on un-notched, notched and welded test pieces of chromium-molybdenum and chromium-molybdenum-vanadium grades of steel, and, in the case of the higher temperatures studied, on chromium-nickel austenitic steels. No attempt is made to discuss the results presented in relation to the properties of a particular steel. The data selected are reviewed only in relation to the light they throw on the following general problems: the amount of scatter which might be expected in different melts of the same grade of material; the relationship between scatter and the safety factor; structural changes during creep testing; the feasibility of determining long-time properties from short-time tests.

Use of Austenitic Steels for Power Plant

A. ENGL: 'The Use of Austenitic Steels in the New Power Station at Hüls.'

Mitteilungen der Vereinigung der Grosskesselbesitzer, 1958, No. 55, Aug., pp. 255–64.

In November 1956, a new power station (operating under initial steam conditions of 300 atm. and 600°C. and, in the reheat stages, under conditions of 140 atm. and 560°C.) was put into operation at the Hüls Chemical Works: an illustrated description of plant design, etc., is given by NOETZLIN, *ibid.*, pp. 230–55. The use of such high temperatures and pressures necessitated, and to a great extent was rendered feasible by, extensive use of austenitic stainless steels. This paper gives particulars of the types of steel employed, discusses the factors which governed the selection of a steel for a specific application, and outlines the problems encountered during operation of the plant and the methods by which they were overcome.

Four grades of niobium-stabilised steel were employed: a chromium-nickel steel, a boron-containing and a precipitation-hardenable vanadium-containing chromium-nickel-molybdenum grade, and a chromium-nickel-molybdenum steel. The first three steels were of 16–13 type; the last was of 16–16 grade. Comparative data on the creep behaviour of the 16–13-type materials in the pertinent temperature range are briefly discussed, and individual components for which each of the four steels was employed are listed in tables relating to the steam boiler, superheater, and turbines.

Economic as well as technical factors governed choice of a particular material. To avoid the high thermal stresses which would have resulted from the excessive wall thicknesses needed with ferritic tubing, austenitic steels were, for example, employed for boiler tubing even at temperatures much below 565°C. (normally considered to be the upper limit for ferritic steels), and economic considerations ensured the use of the vanadium-containing precipitation-hardenable steel for many of the applications for which, on the basis merely of mechanical strength and not of wall thickness, the ordinary 16–13 grade tubing would have proved suitable. Since the precipitation-hardenable steel is subject to severe scaling at temperatures above 650°C., the maximum temperature of the boiler wall was limited to 640°C. None of the austenitic steels was used in cast form.

Before use, specimens from each heat of steel were tested for susceptibility to intergranular corrosion. The procedures used in fabricating, and particularly in welding, the tubes are outlined. Welded or cold-worked tubes of larger diameter were heat-treated as a precaution against stress corrosion. Severe stress-corrosion cracking did, however, occur in the vicinity of the welds when, after 3000 hours' operation, caustic soda penetrated into the boiler feedwater as a result of a fault in the preparation plant. The damage caused is discussed by the authors in some detail, and the type of cracking is illustrated by photomacrographs and photomicrographs of the equipment attacked (*see, in this connection, following abstract*). In many cases the cracks were indiscernible, and corrosion tests had to be carried out, using an experimental boiler, to define the temperature range within which damage could have occurred.

The paper includes short notes on the failure, due to thermal shock, of the jet head of an austenitic-steel water-injection device, and ends with a reference to a failure illustrating the vital importance of ensuring that no notches are present in weldments.

See also following abstract.

Influence of Composition and Microstructure on Stress Corrosion of Precipitation-Hardenable Austenitic Steel

E. BAERLECKEN and K. LORENZ: 'Stress Corrosion and Constitution of the Chromium-Nickel Austenitic Steel X8 Cr Ni Mo V Nb 16/13.'

Mitteilungen der Vereinigung der Grosskesselbesitzer, 1958, No. 54, June, pp. 215-9.

The investigation reported was initiated as a result of severe stress-corrosion cracking which occurred, due to contamination of the feed-water by caustic soda, in welded niobium-stabilised austenitic-steel tubing used in high-pressure power plant (*see previous abstract*). The cracks formed almost exclusively in the vicinity of the welds. It was noted, however, that tubing located at the front and rear ends of the boiler had suffered no damage, and that at the side walls, where cracking did occur, the relative number of cracks appeared to be governed by the location of the weld in the boiler. The tubes at the side walls had been fabricated from different heats of the same grade of precipitation-hardenable steel (nominal composition: carbon $<0{\cdot}1$, chromium 16, nickel 13, molybdenum 1·1–1·5, vanadium 0·6–0·8, nitrogen 0·1, niobium $>10 \times \%$ carbon $+ 4\%$). A statistical analysis of the influence, on susceptibility to stress corrosion, of variation in the composition of the tube and location of the weldment in the boiler indicated that the degree of susceptibility was dependent on the location of the tube, and, for a given location, was related to the heat of steel used for the tube. In the work described the authors have attempted to determine the influence of composition and microstructure on the steel's susceptibility to stress corrosion.

The two heats of steel in question differed to a significant degree only in nitrogen content. All the tubes were therefore classified according to their nitrogen content and their location in the boiler, and a statistical analysis was carried out to relate this classification to susceptibility to attack. The results of this analysis, presented graphically in the paper, show that susceptibility to stress-corrosion cracking is considerably reduced when the nitrogen content is higher than about 0·11 per cent. A similar study of the niobium /carbon relationship led to no conclusive findings.

The authors' attempt to relate microstructure to susceptibility to cracking proved unsuccessful: the often widely differing microstructures exhibited by the cracked tubes (the result mainly of variation in the stabilisation ratio, the nitrogen content and heat-treatment) did not influence crack behaviour.

Corrosion testing of cracked and undamaged tubes (in boiling 65 per cent. nitric acid) threw no further light on factors influencing stress corrosion, but enabled the authors to evaluate, as a function of heat-treatment, slight microstructural changes which were not detectable by optical microscopy.

Spectral-Reflectivity and Solar-Absorptivity Data on Nickel-containing Materials

O. H. OLSON and J. C. MORRIS: 'Determination of Emissivity and Reflectivity Data on Aircraft Structural Materials.'

Wright Air Development Center, Tech. Report 56-222, *Part II, Supplement I*, Oct. 1958; 31 pp.

Total normal emissivity data are presented for the following groups of material which could not be measured in the main test programme, and for which values were not therefore previously recorded in Part II of the report: graphite, magnesium oxide, molybdenum coated with 'Rokide A', plastic laminates, and titanium alloys.

The supplement also contains spectral-reflectivity data (covering wavelengths in the range 0·3 to 2·7 microns) and solar-absorptivity values for chromium, Armco iron, 'AM-350' and A.I.S.I. Type 321 chromium-nickel stainless steels, molybdenum coated with 'Rokide A', 'Inconel X' (in the as-received, cleaned, polished or oxidised conditions), 'Hastelloy B' and three titanium alloys.

Corrosion of Condenser and Heat-Exchanger Tubes

P. T. GILBERT: 'Some Factors Affecting the Performance of Condenser and Heat-Exchanger Tubes.'

Chemistry and Industry, 1959, No. 28, July 11, pp. 888-95.

Although corrosion of condenser tubes is not the serious problem it once was, certain difficulties yet remain to be overcome. In the first part of the paper the author outlines some of the types of corrosion to which condenser and heat-exchanger tubes are subject, and, in each case, refers to the alloys which experience has shown to be the most suitable materials of construction. The final part is concerned with consideration of the performance of tubes from the point of view of operational efficiency.

Reference is made below to salient features of the first part of the paper.

1. Corrosion by Cooling Water

If fresh water is involved, no serious corrosion problems are likely to arise. Use can be made of copper, 70-30 brass, or Admiralty brass (70-29-1). If the water is brackish or seawater and, for an appreciable proportion of the running time, contains dissolved material in amounts higher than about 2000 p.p.m., it is advisable, because of risk of impingement attack, to use a 76-22-2 aluminium brass or a cupro-nickel (usually of 90-10 or 70-30 type).

(a) Impingement Attack

Cupro-nickel alloys exhibit relatively poor resistance to impingement attack unless they contain suitable additions of iron and manganese. That tube makers and users should recognise this fact is emphasised.

(b) Cathodic Protection in Water Boxes

Since the presence of iron in cupro-nickels appears to contribute to the development of a strong self-

healing surface film, it is suggested that corrosion of cast-iron water boxes (which provide a source of beneficial iron corrosion products and, at least initially, some measure of cathodic protection to tube plates and tube ends) might best be minimised by using cathodic protection in conjunction with corroding iron anodes.

(*c*) *Dezincification*

In this connection the necessity of using alpha-brass, suitably inhibited by small additions of arsenic, is emphasised.

(*d*) *Polluted Waters*

No copper-base alloy is entirely immune from corrosion due to water pollution. General experience has, however, shown that the cupro-nickels exhibit the highest corrosion-resistance. Admiralty brass sometimes gives good results with estuarine waters, but aluminium-brass is liable to suffer deep pitting and is not normally recommended for use in contact with polluted waters. Reference is made to the beneficial effects, on corrosion-resistance, of exposing the tubes, before service, to clean water sufficiently long to build up a protective surface film, and mention is made of the promising results obtained by use of the Taprogge system, which ensures continuous removal of harmful scales and deposits from the tubes.

(*e*) *Sand Erosion*

Experience has demonstrated the excellent resistance to sand erosion of a copper-base alloy containing nickel 30, iron 2, manganese 2, per cent. A high tin-bronze (12 per cent. tin) also exhibits good resistance to such conditions.

(*f*) *'Hot-Spot' Corrosion*

is defined as the intensive local pitting that sometimes develops on the water side of the tube, at points corresponding to localised high temperatures at the outside of the tube. Research has shown that, of the materials of interest in this respect, aluminium-brass and 90-10 cupro-nickel exhibit most resistance to such attack.

2. **Corrosion in Steam Spaces**

(*a*) *Incondensable-Gas Corrosion*

occurs as a result of the accumulation of gases to form stagnant pockets, and is more common in such equipment as distilled-water evaporators, sugar evaporators, etc. The main remedial methods are the provision of adequate vents to remove the gases. Of the usual alloys, the 70-30 cupro-nickel is the most resistant to this type of attack.

(*b*) *Use of Copper*

or (where greater strength is required) of 90-10 cupro-nickel tubes has eliminated the corrosion problems associated with 70-30 and 80-20 cupro-nickel tubes in high-pressure feed-water heaters operating under intermittent conditions.

3. **Corrosion by Petroleum Products**

In cases where petroleum products are present on both sides of the tubes mild steel is frequently used. Non-ferrous tubes are, however, employed when water is present on one side of the tube. Aluminium-brass is widely used in oil-refinery service, partly because of its good resistance to corrosion on the water side, and partly because in general it shows reasonably good resistance to corrosion by a wide range of petroleum products. Admiralty brass is, however, preferred by some operators. For acid conditions, or where conditions on the water side make its use preferable, 70-30 cupro-nickel offers advantages.

The main preventive measures against corrosion in oil-refinery service, however, involve suitable modification of the service conditions.

In the second part of the paper the author discusses the importance, in relation to the operational efficiency of the condenser or heat-exchanger, of ensuring that the velocity of the water is the same in each tube, that where necessary the tube is frequently cleaned, and that uniformity of conditions obtain throughout the tube bundle on the steam or product side.

Dislocation Interactions in Chromium-Nickel Stainless Steel

M. J. WHELAN: 'Dislocation Interactions in Face-Centred Cubic Metals, with Particular Reference to Stainless Steel.'

Proc. Royal Soc., Series A, 1959, vol. 249, Jan. 1, pp. 114-37.

The object of the paper is 'to analyse in detail, theoretically and experimentally, the arrangements and interactions of both whole and partial dislocations in face-centred-cubic metals, with particular reference to stainless steel, with a view to understanding the processes which lead to the formation of networks in this material during deformation'.

Annealed foils (0·2 mm. thick) of 18-8-type austenitic steel were deformed by rolling or in tension, and the interaction of dislocations on different slip systems was then studied by transmission electron microscopy and diffraction techniques. Interactions were most frequently observed in piled-up groups, as a result of attractions between the piled-up dislocations and isolated dislocations on other systems. The combination of dislocations took place over short lengths only, leading to the formation of short segments of the resultant dislocation and of networks. The interactions are discussed theoretically, first in terms of undissociated dislocations and then in terms of ribbon dislocations.

Interesting features were found to occur at the nodes of the interacting and resultant dislocations: some nodes were contracted, others were extended into wide regions of fault. In some cases the structures of nodes were resolved, enabling direct comparison with theory. The agreement between theory and observations are considered to verify the concepts of dislocation theory.

Stability of Austenitic Stainless Steels at Low Temperatures

R. P. REED and R. P. MIKESELL: 'The Stability of Austenitic Stainless Steels at Low Temperatures as Determined by Magnetic Methods.'

Proc. 1958 *Cryogenic Engineering Conference*, pp. 84-99; disc., p. 100.
Published Jan. 1959.

The stability of the different grades of chromium-nickel austenitic steels which comprise the A.I.S.I. 300 series has been shown to be critically dependent on chemical composition, and since the nominal composition limits laid down for the series are wide, the M_s temperature may vary considerably in different heats. Cold-working and sensitisation are also known to decrease the low-temperature stability of austenite. In this paper the authors report the results of tests carried out to determine the effects, on the austenite stability of such steels, of operational conditions of importance in low-temperature service.

The steels tested (A.I.S.I. Types 302, 303, 304, 308, 309, 310, 316, 321 and 347) were selected as representative of the various compositions covered by the 300 series, i.e., the 18-8, 25-20, 18-8 Mo, and titanium- or niobium-stabilised grades. The relative stability of the austenite was determined by measurements of magnetic permeability.

In preliminary experiments, designed to establish the maximum permeability of the steels at low temperatures, and conducted mainly on specimens of Type 304 (the most unstable of the grades tested) account was taken of the influence of cold-working plus annealing, thermal cycling between 300° and 76°K. (27° and −197°C.), and of soaking in liquid nitrogen with intermittent cycling (once or three times per day) between the temperature of liquid nitrogen and room temperature. These tests then provided a guide to the procedure necessary to obtain maximum martensite formation.

In the main test programme specimens of each grade were annealed, soaked at 195°, 76° and 20°K. (78°, −197° and −253°C.) and cycled between these temperatures and 300°K. (27°C.), Types 302, 303 and 304 for approximately eight months, the remainder for one month.

The authors' summary of their conclusions is quoted below:

'(1) Commercial 303 and 304 stainless steels can become unstable when soaked at 195°, 76° and 20°K. (−78°, −197° and −253°C.), while the other steels tested can remain stable when soaked at these temperatures and cycled between these temperatures and 300°K. (27°C.).

'(2) The effect of thermal cycling is to increase the rate of martensitic transformation and to increase the total amount of transformation for 303 and 304 stainless steels.

'(3) Annealed 303 and 304 stainless steels tend to transform more on soaking at a low temperature than do 25 per cent. cold-worked samples.

'(4) Even after soaking and cycling for eight months at 76°K. (−197°C.) transformation can still take place in samples of 303 and 304.

'(5) Soaking samples of 304 in liquid nitrogen and cycling between 76°K. (−197°C.) and room temperature produces greater martensitic transformation than does a similar procedure at 20° or 195°K. (−253° or −78°C.).

'(6) The permeability values obtained are considered a maximum for the particular samples tested, if the effects of carbide precipitation and severe cold-working are omitted.'

Low-Temperature Properties of Nickel-containing Materials

See abstract on p. 287.

Wear-Resistance of Austenitic Stainless Steel in Liquid Nitrogen

D. W. WISANDER and R. L. JOHNSON: 'Wear and Friction in Liquid Nitrogen with Austenitic Stainless Steel having various Surface Coatings.'

Proc. 1958 *Cryogenic Engineering Conference*, pp. 71-83.
Published Jan. 1959.

Cryogenic fuels, oxidants, and inert working fluids play an important rôle in propulsion systems for missiles. The bearings, seals, and other parts of turbo-pumps are often lubricated by the liquids which they handle, and, in view of the poor lubricating characteristics of cryogenic fluids of most interest in this respect, it is of obvious advantage to use compatible slider materials. The 18-8-type chromium-nickel austenitic steels (which, because of their suitable low-temperature mechanical properties, form the principal materials of construction used in cryogenic engineering) are, however, susceptible to surface galling or welding during sliding. The investigation described by the authors was therefore undertaken to determine the influence, on the wear and friction characteristics of stainless steel in liquid nitrogen, of various types of surface treatments.

Stainless steel of A.I.S.I. Type 304* was selected for study, and the following surface treatments were used: diffusion coating with nitrogen, sulphur or chromium; chromium electroplating; application of lubricating surface coatings, utilising molybdenum disulphide, polytetrafluoroethylene (PTFE), and polytrifluorochloroethylene (PTFCE).

Wear and friction characteristics were determined using a hemispherical rider specimen sliding, at a surface speed of 2300 ft./min. (690 m./min.) and a load of 1000 gm., in a circumferential path on the flat surface of a rotating disc submerged in liquid nitrogen. The coatings were applied to the disc specimens. Rider specimens were fabricated either

* Carbon 0·08 max., silicon 1·0 max., manganese 2·0 max., phosphorus 0·045 max., sulphur 0·03 max., chromium 18-20, nickel 8-12, per cent.

from Type 304 stainless steel or from a commercial carbon used for sliding contact seals.

Fused spray coatings, 0·01-in. (0·25-mm.) thick, of both PTFE and PTFCE exhibited lower friction and longer endurance life with Type 304 rider specimens than any of the diffusion coatings and most of the solid-lubricant films. Similar results were obtained with a PTFE-glass-filled tape, use of which, tended, however, to be more complicated than that of fused spray coatings. The endurance life of fused PTFE and PTFCE coatings increased with increase in film thickness to the maximum thickness studied (0·01-in.; 0·25-mm.).

With stainless-steel rider specimens sulphurising resulted in lower friction and wear than nitriding, chromising or chromium plating. Both sulphurising and nitriding inhibited welding and metal transfer. Wear of carbon was significantly reduced by sulphurising, chromium plating or nitriding the mating stainless-steel surface, but chromising resulted in increased wear. Friction associated with sulphurised surfaces was similar to that with chromium-plated surfaces, but was much less than with nitrided or chromised specimens.

Wear and friction with carbon specimens riding on specimens coated with resin-bonded molybdenum sulphide and with PTFE-filled porous bronze were low.

Reactor Material Specifications

'Reactor Material Specifications.'
U.S. Atomic Energy Commission, Oak Ridge National Laboratory, Report TID-7017, Oct. 29, 1958; 246 pp.
Compiled and edited by R. M. EVANS.

The materials, welding and inspection specifications contained in the report are 'a culmination of the efforts of the Metallurgy Division of the Oak Ridge National Laboratory to develop methods of ensuring adequate standards for a high-temperature, highly-stressed, experimental nuclear reactor in which reliability of operation was a prime consideration'. Welding and inspection requirements were established in order that most skilled operators could be trained to qualify.

The materials to which the specifications relate are indicated below:

Material Specifications

Inconel: tubing; seamless pipe; plate for hot-forming applications; sheet or strip or tubing for expansion-joint bellows; fittings; hot-formed bar.

Hastelloy B: tubing; welded pipe; plate and sheet; rod and bar.

INOR 8: seamless or weldrawn tubing, precision cold-drawn.

Austenitic Stainless Steel: tubing; pipe.

Welding Specifications

D.C. Inert-Arc Welding: 'Inconel'; 'Inconel' to stainless steel (A.I.S.I. Types 304, 316, 321 and 347); nickel and low-carbon nickel; nickel and low-carbon nickel to 'Inconel'; 'Hastelloy B' and 'Hastelloy C'; carbon steel; Types 304, 316, 321 and 347 stainless steels; 'INOR-8'.

Metal-Arc Welding: 'Inconel' plate; carbon steel.

Inspection Specifications

These specifications relate to inspection of welds, tubing, plate, thick sections of hot-pressed beryllium, piping systems, and formed parts.

The thickness of the materials covered varies, according to the requirements of the individual specification, from 0·1 in. (2·5 mm.) or less to 0·75-in. (18·5-mm.).

A fourth section of the report contains a procedure specification for the joint design and welding of austenitic stainless steel and nickel-base alloys of 0·1-in. (2·5-mm.) thickness or less.

Influence of Irradiation on the Properties of Stainless Steel

W. O. SCHAFFNIT: 'Radiation Damage Studies of Boron Stainless Steel: Final Report.'
U.S. Atomic Energy Commission, Report IDO-16502, Jan. 28, 1959; 27 pp.

The report presents the results of irradiation tests on tensile specimens of an 18-8-type austenitic stainless steel containing 1 per cent. of boron. The investigation was undertaken to determine the changes in dimensions and mechanical properties which might be expected to occur in stainless-steel control rods as a result of increase in neutron exposure during service in power reactors.

After irradiation the specimens were subjected to tensile and hardness tests and to metallographic examination. The data presented show that the material becomes extremely brittle after a relatively short irradiation period. Upon etching, metallographic specimens taken from test pieces irradiated to 25 per cent. burn-up and above were subject, in direct relationship to the amount of radiation they received, to pitting. Evidence of dimensional change with irradiation was not conclusive.

Report of the Corrosion Committee of B.I.S.R.A.

'Sixth Report of the Corrosion Committee.'
Iron and Steel Inst., Special Report No. 66, 1959; 217 pp.
Compiled, on behalf of the Committee, by J. C. HUDSON. Price £3 3s. 0d.

The Fifth Report of the Corrosion Committee (then a Joint Committee of the Iron and Steel Institute and the British Iron and Steel Federation) was presented in May 1938. The present report of the Committee (which is now associated with the British Iron and Steel Research Association) reviews development work since that date. The report is of such proportions as to preclude more than skeleton abstraction, and the greater part of the information given relates primarily to unalloyed steels or to steels alloyed with elements other than nickel. In view, however, of the report's basic interest in connexion

with corrosion research, the headings and sub-headings of the five sections are given below, by way of indicating their scope.

(1) *Development of the Committee's Works Since* 1938.
Constitution; Atmospheric Corrosion; Corrosion by Industrial Waters; Marine Corrosion; Soil Corrosion; Protective Coatings; Corrosion-Test Procedures; Service to Industry; Fundamental Research at Cambridge.

(2) *Atmospheric-Corrosion Tests.*
Introduction; General Experimental Procedure; Tests on Bare Irons and Steels; Effect of Climate on Atmospheric Corrosion; Tests on Painted Irons and Steels; Paint Trials on Iron or Steel Stands; Conclusions.

(3) *Atmospheric Corrosion and Protection at Elevated Temperatures (up to* 300°*C.).*
Introduction; Experimental Programme; Assessment of the Corrosive Conditions; Tests on Different Materials in the Bare Condition; Tests on Protective Coatings; Laboratory Tests; General Conclusions.

(4) *Marine Corrosion Service Trials.*
Introduction; Surface Preparation for Painting; Comparison of Anti-Corrosive Compositions; Service Experience; Conclusions.

(5) Terms of Reference; Acknowledgments; Publications.

Recent Developments in Materials Resistant to Atmospheric Corrosion

E. B. FRIEDL, L. J. NOWACKI and W. H. SAFRANEK: 'Recent Developments in Materials and Finishes to Resist Atmospheric Corrosion.'
Amer. Soc. Mechanical Engineers, Preprint 59-*MD*-8, 1959; 10 pp.

The review, which is based on a *bibliography* of 33 items, directs attention to developments in materials and coatings used to combat atmospheric corrosion resulting from chemical fumes, sea-spray, and moisture.

A section on inorganic coatings (in which the factors discussed include coating vehicles, emulsion-type coatings, pigments and additives, and preparation of metal surfaces), is followed by one on electrodeposited coatings. Bright crack-free chromium plating, duplex nickel plating, and copper-tin alloy plating are assessed in relation to improvements in corrosion-resistance, and notes are given on bonding problems, conversion coatings and anodising. In the remainder of the paper reference is made to literature reporting developments in vapour-deposited coatings, metal-spraying, hot-dipped coatings and in metal-powder coatings, and evaluating the corrosion-resistance of various metals and stainless steels to marine and tropical environments. The paper also contains brief comments on materials (e.g., stainless steel) employed in curtain-wall construction.

Recent Developments in Materials Resistant to Chemical Corrosion

S. W. SHEPARD: 'Latest Developments in Materials to Resist Chemical Corrosion.'
Amer. Soc. Mechanical Engineers, Preprint 59-*MD*-7, 1959; 13 pp.

It is emphasised that the developments discussed relate mainly to improvements in the properties, form, availability or use of established corrosion-resisting materials, and that few new materials have been evolved. The author's notes on such developments are supported throughout by references to the literature (the review is based on a *bibliography* of 100 items). The following fields are covered: aluminium and its alloys; organic coatings; S.G. iron; non-ferrous alloys; plastics; elastomers; stainless steels; titanium and its alloys; uranium and zirconium alloys; miscellaneous materials.

Influence of Structural Factors on Corrosion of Stainless Steel

N. A. NIELSEN: 'The Influence of Structural Factors on the Corrosion of Stainless Steel.'
Werkstoffe u. Korrosion, 1959, vol. 10, July, pp. 429–42.

The data reviewed by the author were derived from corrosion tests designed to establish the influence, on the corrosion-resistance of chromium-nickel stainless steel, of the following variables: composition, heat-treatment, microstructural factors, and the corrosive conditions to which the steel is exposed.

Notes on the phenomenon of passivity, and a discussion of its significance in determining corrosion-resistance and the type and degree of attack to which a specific stainless steel is subject in a given corrosive environment, serve as an introduction to the first, and main, part of the paper, that concerned with factors influencing susceptibility to intergranular corrosion. The information given relates to steels of 18-8 and 18-8-Mo grade (A.I.S.I. Types 304 and 316), the corrosion characteristics of which are illustrated by reference to a series of photomicrographs indicating the type and progression of corrosion suffered by the materials, in the annealed and in the sensitised conditions, when exposed to nitric-acid, chromic-acid, sulphuric-acid and hydrochloric-acid solutions.

That the sensitised specimens of the 18-8-Mo grade were more severely attacked in boiling 65 per cent. nitric acid than the sensitised molybdenum-free type might, it is tentatively suggested, be attributed to the presence of sub-microscopic ferrite precipitates, or of sigma phase, in the former steel. Further experiments using the same corrosive medium were carried out on specimens heat-treated to induce precipitation of delta ferrite at the grain boundaries. The Type 304 steel suffered no significant intergranular attack either in the sensitised or annealed condition, but the specimens tested were coarser grained than the ferrite-free test pieces. General corrosion in both conditions of heat-treatment was similar.

Corrosion of annealed specimens of the dual-phase 18-8-Mo steel was low and was predominantly of a general nature: intergranular attack of sensitised test pieces was, however, severe and the type of attack resembled that of the single-phase steel. This resemblance leads the author to suggest that a fine sub-microscopic precipitation of sigma phase might be present in standard grades of 18-8-Mo steel, and that the high corrosion rate exhibited by the steel after sensitisation might be attributed to this precipitate. It is considered likely that the form and distribution of ferrite, as well as of sigma phase, play an important rôle in the corrosion behaviour of the sensitised 18-8-Mo steel. The finer the distribution of ferrite or sigma phase in the grain boundaries, the greater is the area available for carbide precipitation, and hence the severer is the intergranular attack. All other conditions being equal, corrosive attack on a ferrite/austenite/carbide surface should be a function of the size and distribution of the ferrite (or sigma phase) exposed, at the grain boundaries, to the effect of the nitric acid. The remainder of this sub-section is concerned with discussion of photomicrographs illustrating progressive corrosion of annealed or sensitised specimens of the two austenitic steels in boiling 10 per cent. chromic acid, boiling 3 per cent. sulphuric acid, and boiling 3 per cent. hydrochloric acid.

The influence of grain-size on intergranular corrosion was investigated on coarse- and fine-grained specimens of the 18-8 type steel, in both the sensitised and the annealed condition. Corrosion data derived from tests in the standard boiling-nitric-acid solution and (at 70°C.) in a 10 per cent. nitric-acid/3 per cent. hydrofluoric-acid solution are presented. The grain-size of annealed specimens was found to have no significant influence on the corrosion rate in the nitric-acid solution, but, in the sensitised condition, the degree of corrosion suffered by the coarse-grained material was considerably less than that suffered by the fine-grained; rate of penetration was similar in both cases, but, even after 600 hours' exposure, disintegration occurred in the coarse-grained material only in the smallest of the grains. The higher corrosion-resistance exhibited by the sensitised coarse-grained steel is attributed to the longer time required to undermine the grains of this material. In tests with 10 per cent. chromic acid, and 10 per cent. nitric-acid/3 per cent. hydrofluoric-acid solutions similar results were obtained.

In the next sub-section attention is directed to the influence, on intergranular corrosion of austenitic steel, of the relative orientation of neighbouring grains. The effects of grain-orientation in this respect are briefly explained in terms of atom disorder, and are illustrated by reference to photomicrographs showing intergranular attack on twinned grains.

In the final sub-section of those concerned with intergranular corrosion the author reports the results of experiments carried out to determine the effects of cold-working on the corrosion-resistance of 18-8 steel. The data presented (derived from tests, in boiling nitric acid, on specimens cold-worked before and/or after sensitisation) show that cold-working to an 80 per cent. reduction results in an appreciable improvement in corrosion-resistance. In assessing the corrosion data it is emphasised, however, that account must be taken of such variables as the form and distribution of the carbides precipitated during sensitisation, the formation of ferrite during cold-working, etc.

Pitting corrosion of stainless steel is discussed mainly in relation to investigations by STREICHER, which indicated, both in simple immersion and in electrolytic accelerated tests, that grain boundaries rather than non-metallic inclusions are the primary sites on which pitting starts.

The paper ends with a short *résumé*, with photomicrographs, of the results of metallographic investigations of the effects, on the structure of stainless steel, of high-temperature oxidation and thermal attack.

Intergranular Corrosion of Stainless Steels: Determination of Time/Temperature/Sensitisation Diagrams

H. F. EBLING and M. A. SCHEIL:

(1) 'Corrosion-Resistance of Type 347 Stainless after 'Sensitising' Heat-Treatments.'

Metal Progress, 1959, vol. 76, July, pp. 94-7.

(2) 'TTS Diagrams for Types 304L and 316L Stainless.'

Ibid., 1959, vol. 76, Aug., pp. 87-91.

In their introduction to the two parts of the paper the authors briefly discuss intergranular corrosion of stainless steel in relation to sensitisation, carbide and sigma precipitation, and the efficacy of the various laboratory corrosion tests in determining susceptibility to such attack. The degree of sensitisation is governed to a great extent by the temperature to which the steel is exposed and the time of exposure, and the investigation described was carried out with the aim of establishing the effects of these two factors on the resistance of different grades of chromium-nickel stainless steel to intergranular corrosion.

The stainless-steel specimens were aged for times up to 10,000 hours at temperatures ranging, at intervals of 100F°. (56C°.), from 700° to 1500°F. (370° to 815°C.). Resistance to intergranular corrosion was evaluated by the boiling-nitric-acid test. The specimens were immersed in the acid for five 48-hour periods, except when the corrosion rates exceeded 1 in./year. Based on the data obtained (and using the time/temperature relationship for any particular corrosion rate) a graph can be constructed indicating the corrosion rates which result from heating the steel for a given time at a given temperature. A graph of this type is somewhat similar to a TTT diagram, and is in this paper designated a TTS (time/temperature/sensitisation) diagram.

In the first part of the paper TTS diagrams and photomicrographs are presented for two commercial heats of A.I.S.I. Type 347 niobium-stabilised 18-8

stainless steel, in the mill-annealed condition and after a stabilisation heat-treatment. The diagrams discussed in the second part were determined for heats of low-carbon 18–8 chromium-nickel and 18 – 12 – 3 chromium – nickel – molybdenum steels (A.I.S.I. Types 304L and 316L) in the mill-annealed condition.

It is shown that mill-annealed sheet of Type 347 steel can be sensitised by heating at temperatures above 900°F. (480°C.) and reaches a maximum degree of sensitisation at a characteristic time; prolonged exposure at temperature then induces true stabilisation. A prior stabilisation heat-treatment of 2 hours at 1650°F. (900°C.) considerably increases the corrosion-resistance of the steel. The data obtained for Type 304L steel indicate that the material is resistant to sensitisation during normal short-time fabricating heat-treatments. This steel, however, offers no other advantage over Type 347 steel, and, in the light of present knowledge, should not be substituted for the latter steel since it cannot be stabilised by heat-treatment; Type 347, on the other hand, is little better than Type 304L if used in the commercial annealed condition. The curves in the TTS diagram for Type 316L steel lie about 100F.° (56C.°) above those for the other two grades. They are closely packed and lie much further to the left, indicating that the heating schedules which cause corrosion rates higher than 1 in./year are shorter: less than one hour at temperatures in the range 1300°–1400°F. (705°–760°C.).

The TTS diagrams so far determined differ according to the type of steel to which they relate, and it is expected that if, as a result of further work involving tests on many more heats of material, these differences are established as characteristic, the diagrams will provide the engineer with fundamental corrosion data.

Influence of Environment on Stress Corrosion of Stainless Steel

L. E. PHILLIPS: 'Autoclave Testing of Type 304 Stainless Steel.'

U.S. Office of Technical Services, Army Report MND-E-1322, May 1958; 54 pp.

The investigation was initiated due to concern over the possibility of stress-corrosion failure of 18–8-type chromium-nickel stainless steel (A.I.S.I. Type 304) intended for use in the steam generator of the Army Package Power Reactor. It was postulated that the greatest risk of stress corrosion was in the crevice area of the superheater tubing, where cracking could be induced as a result of build-up of secondary-water chlorides and the presence of residual stresses in the tubing. The aim of the investigation was to define the maximum amounts of impurities which, under the temperature and pressure conditions obtaining in the secondary system of the reactor, could be tolerated in the water without risk of stress-corrosion cracking. The data obtained were then to be used to determine the environmental conditions for operation of pilot heat exchangers.

U-bend specimens of Type 304 stainless steels were exposed, in rocker autoclaves, both to the vapour and the liquid phase of the corrosive-water environment. In varying the chemistry of the water to which the specimens were exposed, a study was made of the influence of the pH and of the chloride, phosphate and oxygen contents of the water. The experiments were prepared on a statistical basis: three levels of control were assigned to each variable (e.g., the pH was adjusted to 8·5, 10·4 or 11·5), and the control levels were randomly distributed among the tests. After addition of the desired amounts of chloride and phosphate, the test water (tap-water, distilled water or de-ionised water) was aerated, boiled, or oxygenated to achieve the required oxygen content. The oxygen content of the atmosphere above the surface of the water in the autoclave was also varied.

From the data obtained (which are supplemented in the report by numerous photomicrographs correlating cracking with environment) it is concluded that the presence of oxygen and chlorides in the water was the only factor which contributed to the cracking of test specimens. The chloride content at which cracking was induced was governed by the duration of the test, while the critical oxygen level appeared to be independent of the time of testing. The results indicate, however, that at a high oxygen level cracking does not occur if the chloride concentration is low, and *vice versa*, a finding which is in contradiction to some reports in the literature. The oxygen level necessary to induce cracking was found to be approximately 4 per cent.

The results also indicated that oxide-film formation on stainless steel may be sufficient to induce stress-corrosion cracking in the presence of chlorides, even if the environmental oxygen concentration is low.

The surprisingly high concentration of oxygen which was found necessary to induce cracking is considered to be a most significant finding, since it suggests the possibility that un-treated water can be used without risk of stress corrosion, provided that provision is made for adequate de-aeration of the boiler.

Carbon-steel specimens were subjected to severe environmental conditions without occurrence of cracking. Their general corrosion-resistance was, however, poor.

Stress Corrosion of Stainless Steel in Chloride Waters

R. W. STAEHLE, F. H. BECK and M. G. FONTANA: 'Mechanism of Stress Corrosion of Austenitic Stainless Steels in Chloride Waters.'

Corrosion, 1959, vol. 15, July, pp. 373t–80t; disc., p. 381t.

The purpose of this paper is 'to discuss the effect of the relative availability of chloride and oxygen and the applied stress on the morphology of stress-corrosion cracks in austenitic stainless steels'.

The stress-corrosion tests were carried out, in 550-ml.-capacity autoclaves, on A.I.S.I. Type 304L stainless steel of the following composition: carbon 0·07, silicon 0·61, manganese 1·48, sulphur 0·015, phosphorus 0·018, chromium 17·15, nickel 10·36, niobium 0·97, per cent. The test procedure and apparatus (including the stressing jig) are fully described. Prior to testing all the specimens were held at 2000°F. (1095°C.) for ½ hour and then furnace-cooled. The influence of surface variables was studied by subjecting the specimens to one of the following treatments: belt-abrading with 120 grit; pickling six hours in concentrated hydrochloric acid; electrolytic polishing. Specimens subjected to the first treatment had residual surface stresses, while those given the last two treatments did not.

The sodium-chloride concentration of the test solution was varied in the range 50–80,000 p.p.m. In all cases the autoclave was filled with 200 ml. of solution, but in the experiments concerned with investigation of the effects of various gases on the propensity to stress-corrosion cracking, the air in the remainder of the autoclave was replaced by oxygen, hydrogen or nitrogen.

The specimens were tested both in the water phase and under vapour-condensation conditions. Tests were run for various lengths of time, but cracking for all concentrations and physical conditions appeared to be fully developed after four hours exposure at 400°F. (205°C.).

The data presented in the paper are supplemented by photomicrographs illustrating the path and depth of the stress-corrosion cracks. The results of the investigation are considered by the authors to justify the following conclusions:

'1. Type 347 stainless steels are susceptible to stress-corrosion cracking in the presence of 400°F. (205°C.) chloride-containing waters, when exposed to vapour-condensation conditions. The threshold stress applied for this condition is lower than 2,000 p.s.i. (1·5 kg./mm.²), and chloride concentrations as low as 50 p.p.m. in the water will produce cracking. Increasing the oxygen concentration from that in air to 100 per cent. increases the probability of cracking at low chloride concentrations, while the presence of 100 per cent. hydrogen or nitrogen above the solution eliminates cracking.

'2. Type 347 is susceptible to stress-corrosion cracking under highly specific conditions when immersed in chloride solutions at 400°F. (205°C.). The solution-concentration range of susceptibility is 20,000 to 25,000 p.p.m. of NaCl, and the stresses must exceed 30,000 p.s.i. (13·5 t.s.i.; 21 kg./mm.²).

'3. Type 347 does not crack in a saturated vapour phase (no condensation) above an 875 p.p.m. NaCl solution at 400°F. (205°C.).

'4. Cracking progresses periodically. After the initial pit forms, the crack progresses in short abortive branchings. In the second stage of cracking, the cracks move in longer bursts of 2–10 grains in length. The onset of this stage is probably controlled by a certain stable crack size reached in the first stage. The third stage would be catastrophic if the specimen were under constant load instead of being beam-loaded.

'5. On the basis of the cracking at the low stresses, it is proposed that the actual crack movement results from electrochemical action, with the stress serving to provide a local region of highly stressed metal at the base of the crack. This highly stressed metal provides a local anodic area for preferential electrochemical dissolution.'

Thermal-Cycling Stress-Corrosion Tests on Stainless Steel in Lithium-Hydroxide-containing Water

G. H. BORRMANN and G. E. GALONIAN: 'Results of Thermal-Cycling Stress-Corrosion Tests of Type 347 Stainless Steel in 500°F. pH 10–11 Lithium Hydroxide.' *U.S. Atomic Energy Commission, Report KAPL-M-GHB-1*, Jan. 8, 1959; 12 pp. + tables and figures.

The investigation described was initiated to determine the resistance of stainless steel to primary coolant water containing additions of lithium hydroxide intended to raise the pH to the range 10–11. Under normal operating conditions no corrosion difficulties were anticipated since the hydroxide solution would be dilute, but there was concern that, in some regions of the primary system, stress-corrosion cracking or accelerated corrosion might, due to concentration of the lithium hydroxide, occur in stainless-steel structural materials. Such a concentration would be most likely to occur as a result of (1) boiling in heated crevices, or (2) build-up of deposits by leakage of water to the atmosphere. Corrosion tests were therefore carried out to study the effects of these two conditions. (During the tests now described the specimens were subjected to thermal cycling. The results of tests under steady-state conditions have been reported by GALONIAN in *Report KAPL-M-GEG-11*: see abstract in *Nickel Bulletin*, 1959, vol. 32, No. 1, p. 23.)

Three stressed niobium-stabilised chromium-nickel stainless-steel bolts were exposed, at 371°, 336° and 355°C., respectively, (i.e., at temperatures sufficient to induce boiling) to a lithium-hydroxide solution at 500°F. (260°C.) and were cycled between these temperatures and an equilibrium temperature three times per week for one month. The effects of concentration by leakage were evaluated by stress-corrosion tests on specimens of stabilised and unstabilised 18-8 steels, using a Schroeder embrittlement detector.

Uniform black corrosion films formed on the bolts, but, though a few small fissures were found in the roots of some threads, there was no evidence of accelerated corrosion and no lithium hydroxide or other deposits formed on the heated surfaces. No cracking occurred in the niobium-stabilised-steel embrittlement specimens during two months' exposure, and the unstabilised stainless steel exhibited no unusual corrosion effects as a result of one week's exposure to leakage.

Corrosion of Chromium-Nickel-Molybdenum Stainless Steels in Phosphoric Acid

A. R. MORGAN: 'Corrosion of Types 316 and 317 Stainless Steel by 75 per cent. and 85 per cent. Phosphoric Acid.'

Corrosion, 1959, vol. 15, July, pp. 351t–4t.

The Company with which the author is associated produces white phosphoric acid in wetted-wall furnaces made of standard and low-carbon grades of chromium-nickel-molybdenum stainless steels (A.I.S.I. Types 316 and 316ELC). The acid produced and recirculated in the furnace is usually at a concentration of 75–76 wt. per cent., although 78 and 85 per cent. acids are also produced. Considerable heat is liberated in the production of phosphorus, and, since this heat is absorbed by the acid, it was necessary to determine the maximum temperature at which the acid could be handled without deleterious effects on the equipment. To obtain the necessary data, corrosion tests were carried out on Types 316† and 317* stainless steels. The results of these tests are recorded in the present paper.

The test programme was carried out in three phases:

(1) Laboratory tests using a constant-temperature bath. Specimens were exposed to two concentrations of the acid (76 and 85 per cent.) at temperatures of 85°, 100° and 115°C., and were weighed at 2, 8, and 16 days from the start of each run. Both concentrations of acid were used under two conditions at each temperature: aerated and agitated, and static. Five specimens of Type 316 (the only steel studied in this phase of the programme) were tested, comprising an as-cast and a rolled test piece, a welded specimen (i.e., a rolled specimen with a bead of low-carbon (0·03 max.) grade of Type 316 weld metal laid across the face), and a rolled and a cast specimen placed in direct contact with each other.

(2) Tests, on Types 316 and 317 steels, in 75 per cent. phosphoric acid, at 78°, 85°, 90°, 95° and 105°C., using a pilot apparatus simulating the conditions of the production furnace.

(3) Plant tests on specimens of Type 316, 316ELC, and 317 steels suspended, at temperatures in the range 77°–103°C., at various locations in a plant-operating furnace.

Corrosion rates were found to be considerably influenced by acid temperatures and by testing conditions. Although laboratory data indicated a maximum permissible temperature of over 115°C. for the Type 316 steel in either 75 or 85 per cent. phosphoric acid, and pilot-plant data indicated a maximum permissible temperature higher than 105°C. at an acid concentration of 75 per cent., the extensive data derived from plant tests showed that use of Type 316 and 316ELC steels was acceptable (i.e., corrosion rates were less than 5 mils/year) only at a temperature of 100°C. or less. Type 317 steel proved to be more resistant than Type 316ELC in both the pilot and the plant tests. Samples exposed to an aerated and agitated environment exhibited lower corrosion rates than those in a stagnant test medium. Welded specimens were as resistant as solid sheets in all the tests. The corrosion-resistance of cast specimens of Type 316 was similar to that of as-rolled test pieces.

† Carbon 0·08 max., silicon 1·0 max., manganese 2·0 max., sulphur 0·03 max., phosphorus 0·045 max., chromium 16-18, nickel 10-14, molybdenum 2-3, per cent.

* The composition of Type 317 differs from that of Type 316 as follows: chromium 18-20, nickel 11-15, molybdenum 3-4, per cent.

Corrosion of Nickel-containing Aluminium Alloys in High-Temperature Water

K. VIDEM: 'Corrosion of Aluminium Alloys in High-Temperature Water: A Survey.'

Jnl. Nuclear Materials, 1959, vol. 1, July, pp. 145–53.

Aluminium possesses properties which render it an attractive material for use in water-cooled reactors, and in those operating at relatively low temperatures aluminium canning has been in use for many years. In reactors operating at high temperatures, however, aluminium has two serious disadvantages: its poor resistance to high-temperature water and its low creep strength. Work by DRALEY and RUTHER showed that the corrosion-resistance of the metal could be considerably increased by alloying additions of from 0·5–2 per cent. of nickel (see, for example, abstract in *Nickel Bulletin*, 1958, vol. 31, No. 1, pp. 19–20, in which reference is also made to abstracts relating to previous literature), and research has since continued to improve the properties of such alloys. In the present paper the author reviews the conclusions which can be drawn from work reported in the literature.

The discussion relates mainly to work on three types of aluminium alloy, containing, respectively, nickel 0·5–2, iron 0·2–0·5, silicon 0·2, per cent.; silicon 10, nickel 1, per cent.; and copper 4–8, nickel 1–2, per cent.

A survey is given of test procedures used to evaluate such alloys, and their corrosion behaviour under conditions simulating reactor service is reviewed. The paper also contains brief references to literature concerned with welding characteristics and mechanical properties, but the author's principal concern is with corrosion mechanisms. In high-temperature water the alloys are subject to three types of corrosion: grain-boundary attack; accelerated attack; and uniform attack. Grain-boundary corrosion can be prevented by alloying with elements which form phases which are cathodic to aluminium, and the accelerated type of attack can be controlled by proper fabrication technique and appropriate additions of alloying constituents. Uniform attack is thus the main corrosion problem, and since it is suggested that dissolution of aluminium oxide is the rate-controlling factor in a dynamic system, it is considered difficult to propose alloys with higher corrosion-resistance than the best of those at present available. Property designed, aluminium-clad fuel elements

might, however, be expected to give satisfactory service at 230°C. and perhaps even at somewhat higher temperatures.

Corrosion of Austenitic Stainless Steels in High-Temperature Water Under Heat-Transfer Conditions

J. N. WANKLYN and D. JONES: 'The Corrosion of Austenitic Stainless Steels Under Heat Transfer in High-Temperature Water.'

Jnl. Nuclear Materials, 1959, vol. 1, July, pp. 154–73.

For some types of pressurised-water reactors it is possible to consider cladding the fuel with austenitic stainless steel. This application, however, requires good corrosion-resistance under very high rates of heat-transfer. The experiments described were designed to determine the corrosion-resistance of two stabilised 18–8-type chromium-nickel stainless steels under the heat-transfer conditions to which they would be subjected during such service.

The results of the investigation have been briefly reported by the authors in *Chemistry and Industry*, 1958, No. 28, July 12, pp. 888–9, and the scope of the test programme and the salient findings were indicated in the relevant abstract in *Nickel Bulletin*, 1958, vol. 31, No. 11, p. 302. The present paper contains a detailed account of the experiments carried out and the data obtained.

Corrosion of Nickel-containing Magnesium Alloys

J. H. GREENBLATT: 'Corrosion of Magnesium Alloys containing Iron and Nickel.'

Jnl. Applied Chemistry, 1959, vol. 9, Aug., pp. 401–5.

The experiments reported formed part of research work to determine the feasibility of using nickel- or iron-containing magnesium alloys as materials for soluble plugs in sea-water. Preliminary corrosion tests had demonstrated that the presence of iron or nickel increased the corrosion rate of the magnesium alloy to a degree sufficiently high for this application, but when plugs fabricated from the alloys were tested in the metallic device in which they were to be used, the corrosion rates were found to be markedly lower than expected. To determine if this decrease in the corrosion rate were the result of a passivating mechanism, corrosion tests were carried out in sea-water on magnesium alloys containing 2 per cent. of nickel or 200 p.p.m. of iron.

Before testing the alloys were subjected to one of the following treatments: etching in 0·5 *N*-HCl; exposure to 5 per cent. nitric acid for 5 minutes; and sandpapering. Corrosion rates were then determined for specimens (1) embedded in plastic blocks, or (2) coupled with steel cathodes of various sizes to give a range of cathode/anode ratios.

The corrosion rate of the uncoupled nickel-containing alloy was found to be higher than that of the iron alloy. The initial condition of the specimens had a pronounced effect on the corrosion rate. Pre-treatment by etching with hydrochloric acid greatly increased the rate of corrosion exhibited by both alloys, while pre-treatment with nitric acid resulted in very low initial rates of corrosion. The length of time for which nitric-acid-treated specimens remained inhibited varied between 4 and 16 hours. The presence of hydroxyl ion caused a substantial decrease in the corrosion rate.

The tests on coupled specimens showed that the corrosion rate decreased with increase in the size of the steel cathode, although this size factor was less critical for the magnesium-nickel alloy than for the iron-containing one. These results indicate that the decrease in the corrosion rate of coupled specimens may be attributed to the formation of an oxide film on the surface of the alloys, and data obtained from tests on the alloy/steel couples suggest that the critical current density for formation of the film is 50 mA./cm.^{2}.

Corrosion of Sour-Water Stripping Units

J. F. MASON and C. M. SCHILLMOLLER: 'Corrosion in Sour-Water Strippers.'

Corrosion, 1959, vol. 15, July, pp. 358t–62t; disc., p. 362t.

The efforts of the refining industry to develop an effective method of disposing of sulphide-bearing waters without causing air or water pollution have focussed attention on means of minimising corrosion of the equipment used. In this paper the authors outline the various techniques employed to strip the sour water and to dispose of the waste water and gas, review the corrosion problems encountered in sour-water strippers, and discuss the methods which have been adopted to control corrosion (inhibition, use of non-metallic coatings, and selection of appropriate materials of construction).

The main section of the paper is concerned with presentation and discussion of the results of stripping-plant corrosion tests on the following materials: 'Monel', 'Inconel', 18–8 type and straight-chromium stainless steels, aluminium, mild steel, 'Ni-Resist' and cast iron. The data relate to tests in the vapour phase of a water-reclaimer tower; in a high-pressure water-adsorption system for the removal of acidic gases (such as CO_2 or H_2S) from hydrocarbons; in water vapour containing diethanolamine, H_2S, and traces of NH_3, CO_2 and hydrocarbons; in water separated from light straight-run gasoline; and in 10 per cent. phenolic water vapour containing chlorides.

The corrosion problems associated with any sour-water stripping unit are probably unique, but nevertheless the authors believe that general conclusions on corrosion control can be drawn from the data obtained. In the overhead condenser of the stripper, use of materials such as aluminium, 'Monel' and 18–8–Mo stainless steel have effectively solved the main corrosion problem, but each material has certain peculiarities and shortcomings that should be taken into account before selection. Introduction of such

materials has proved justifiable where corrosion rates exhibited by carbon steel exceeded 25 mils/year.

Use of inhibitors and of 'Gunite' and alloy linings has proved satisfactory in protecting the stripping column, the standard material of construction for which is carbon steel. In general, corrosion can be minimised by improving plant practice (proper operation, and control of the pH of the sour-water feed), by avoiding the most deleterious conditions (high temperatures and pressures, presence of oxygen, velocity effects and impingement), and by selection of suitable materials ('Monel', 18-8-Mo steel, aluminium or 'Gunite').

Corrosion Resistance of Titanium-, Zirconium- and Nickel-containing Materials in Chemical-Plant Environments

P. J. GEGNER and W. L. WILSON: 'Corrosion-Resistance of Titanium and Zirconium in Chemical-Plant Exposures.'

Corrosion, 1959, vol. 15, July, pp. 341t-50t.

The increase in the availability of titanium and zirconium has stimulated interest in the use of the metals as materials of construction in the chemical industry. Most of the relevant corrosion data are, however, derived from laboratory tests and do not provide a basis for comparison with those obtained from materials commonly used commercially for the specific corrosive environment under consideration. The investigation described was initiated with the aim of providing data on the resistance, under service conditions, of commercially pure zirconium and titanium to a wide variety of corrosive media.

Seven chemical plants, most of which fell within the chlor-alkali group, participated in the test programme. Specimens were exposed, wherever possible for at least one month, to the following environments: calcium-chloride solutions, alkali solutions, gaseous chlorine, solutions containing free chlorine, hypochlorite solutions, sulphuric-acid solutions, nitric-acid solutions, hydrogen chloride and hydrochloric acid, salt solutions, chlorine-containing atmospheres, and various other media.

The data obtained for the zirconium and titanium specimens are tabulated in detail, in terms of corrosion rates per year. Comparative data are presented on the following materials, which were exposed under similar conditions to those under which the zirconium and titanium were tested, but not necessarily to all the environments: nickel, 'Monel', 'Inconel', 'Ni-O-Nel', 'Hastelloy B', 'Hastelloy C', 'Hastelloy D', 'Hastelloy F', 18-8-type stainless steels, 'Ampco 8', 'Chlorimet 2' and 'Chlorimet 3', 'Worthite', 'Duriron', 'Durichlor', 'Carpenter 20', 'Incoloy', 'Incoloy 804', 70-30 and 90-10 cupronickels, platinum-plated titanium, aluminium-brass, cast iron, mild steel, lead, aluminium, copper, niobium and tantalum.

Corrosion Tests on Nickel-containing Materials in Plant Processing Tall Oil

H. C. TEMPLETON: 'Corrosion Testing and Corrosion Problems in Processing of Tall Oil.'

Jnl. Amer. Oil Chemists' Soc., 1959, vol. 36, Mar., pp. 127-9.

The author's aim is to present and evaluate corrosion data derived from test specimens exposed in the following plant processing tall oil:
(1) a tall-oil recovery system handling a tall-oil/naphtha + sulphuric-acid solution at 200°F. (95°C.); (2) a high-temperature reactor (involving exposure to successive esterifications, amidisations and sulphurisations of tall oil); (3) and (4) the base of tall-oil-fractionating towers handling, at 265°C., solutions of, respectively, 20 per cent. oleic-linoleic acids/60 per cent. rosin acids/20 per cent. tall-oil pitch and 65 per cent. fatty acids/35 per cent. rosin acids in the liquid phase.

The materials tested (though not necessarily in all four plants) were: Type 304 (18-8) and Types 316 and 317 (18-8-Mo) stainless steels, carbon steel, 'Aloyco 20', 'Worthite', 'Ampco 18', 'Monel', 'Inconel', 'Hastelloy B', 'Hastelloy C', 'Ni-Resist' and nickel.

Except for Type 304 all the stainless steels were resistant to attack by the solutions of plants (1) and (3). Of the specimens exposed in plant (4), only Type 317 stainless steel (which has a higher molybdenum content than Type 316) was not heavily attacked. Type 316 steel satisfactorily withstood the conditions to which it was exposed in plant (2).

In evaluating the data the author concludes that use of Type 316 is necessary in the tall-oil recovery system, though 'Worthite' and 'Aloyco 20' are also suitable for such service. For equipment in distillation and fractionating plant use of Type 316 stainless steel is essential. For severer conditions the higher-molybdenum grade, Type 317, should be employed, and at temperatures higher than 575°F. (300°C.), when this steel becomes unsuitable for use, 'Inconel' and 'Hastelloy C' exhibit satisfactory corrosion-resistance.

Technique for Investigation of Pitting Corrosion of Stainless Steel

J. M. DEFRANOUX: 'Application of the Polarisation Curve to Investigation of Pitting Corrosion in Stainless Steels.'

Werkstoffe u. Korrosion, 1959, vol. 10, July, pp. 425-9.

In a short introductory review of the causes and morphology of pitting corrosion the author divides the factors which lead to such attack into two classes: 'idiomorphic' factors (difference in composition or microstructure of the steel); 'exomorphic' factors, i.e., those pertaining to the fabrication of the steel (cold-working, welding, etc.), or to the specific conditions to which the material is subjected during service (concentration of corrosion products, absence of oxidising media, galvanic corrosion, etc.). The significance of exomorphic factors in relation to

pitting corrosion is discussed. The importance of different 'exomorphic' variables has been frequently emphasised, but in previous studies of pitting corrosion which have involved use of electrochemical techniques (the paper includes a brief review of the relevant literature) little or no account has been taken of their effects.

In the present paper the author describes a laboratory corrosion-testing technique which is stated to permit determination of the possible influence of each of the 'exomorphic' elements involved in specific corrosive conditions, and hence enables prediction of the risk, and type, of pitting corrosion associated with a given application. The stainless-steel specimen is polarised, first anodically and then cathodically, in the relevant corrosive medium. Corrosion rates can then be calculated from the point of intersection of the two polarisation curves. The utility of the technique is illustrated by a description of the procedure used to evaluate the behaviour of 18-8 type chromium-nickel steel under various corrosive conditions.

Surface Finishing of Stainless Steels

L. F. SPENCER: 'Surface Finishing Stainless Steels.' *Metal Finishing*, 1959, vol. 57, July, pp. 48-53 and 58.

The review is in five sections. In the first section the author discusses the three types of stainless steel available (austenitic, ferritic and martensitic) and in the next, emphasising that the degree of surface finishing required to obtain a specific lustre on fabricated parts is frequently related to the surface condition of the purchased material, he defines the mill finishes (for sheet and strip) which have been standardised by the American Iron and Steel Institute. In the third section reference is made to the factors governing selection of a sheet finish for a specific application, and this discussion leads, in the next section, to consideration of the characteristics which necessitate variances in the usual finishing procedures. Finally the author recommends procedures for use in grinding, polishing and buffing stainless steels.

An interesting feature of the article is a table summarising the procedures required to produce each of the nine finishes discussed in the text, and containing recommendations with respect to abrasives, lubricants, and type, size and speed of wheel.

Use of Nickel-Chromium-Boron Hard-Facing Alloys in the Sugar Industry

E. SELLIER: 'Hard-Facing in the Sugar Industry with Alloys containing Chromium Borides.'

Paper printed by *Robert Louis*, Brussels, 1959 ; 8 pp.

Breakdowns in production equipment used in the sugar industry can result in serious loss of sugar, due to deterioration of the beet awaiting processing. Such breakdowns occur mainly in components subjected to corrosion, impact and, in particular, abrasion. In this paper the author, who is associated with S.A. L'Oxhydrique Internationale, Brussels, describes the use which has been made, in a Belgian sugar-producing plant, of hard-facing techniques, as a means of salvaging worn equipment and obtaining the necessary abrasion-resistance.

Factors in favour of hard-facing such equipment are listed, and an equation is presented which permits calculation of the economic advantages or disadvantages of hard-facing (*vis-à-vis* replacement of) a given component. Reference is made to data on pertinent properties of three nickel-chromium-boron-base hard-facing alloys, and in the main section of the paper the use of these alloys in the sugar industry is exemplified by discussion of the techniques employed in, and the advantages derived from, hard-facing the following components: hooks of lifter buckets, shafts and axles of diffusion equipment, and the teeth of grab buckets.

Hard-Facing with Nickel-base Alloys

R. P. CULBERTSON, W. E. NORDEN and J. M. RUZEK: 'Hard-Facing with Alloys of Cobalt and Nickel.'

International Institute of Welding, 1959 *Annual Assembly*, *Preprint*; 7 pp.

The emphasis in the paper is on hard-facing with cobalt-base alloys: of the nine alloys discussed six are of cobalt-chromium-tungsten type, two of nickel-chromium-silicon-boron type, and the last of iron-chromium-tungsten-nickel type.

An introductory section outlining the advantages which hard-facing offers, and discussing the properties required in hard-facing materials (high degree of hardness at normal and elevated temperatures, ability to take and maintain a high polish, and resistance to scoring, galling and corrosion) is followed by a short review of the salient metallurgical characteristics of each alloy. This review includes notes, supplemented by photomicrographs, on as-deposited and heat-treated microstructures, and on the variation of relevant properties (e.g., hot-hardness) with composition and microstructure. The corrosion-resistance of cobalt-base alloys is briefly discussed.

The remainder of the paper is concerned with the procedures used, and factors of importance, in hard-facing with such alloys. In this connection welding and powder-spraying techniques are discussed, and in other sections (on surface preparation and pre-heating, application of the alloys, and machining and grinding the deposits) reference is made, *inter alia*, to the following factors which adversely affect the quality of the deposits: the presence of sharp corners and recesses in the basis metal, dilution in the basis metal, and shrinkage in the deposit. Careful preparation of the surface and use of correct procedures in depositing the alloys will minimise or reduce dilution, while shrinkage can be reduced by proper post-heat control.

Diffusion-Bonding Characteristics of 'Zircaloy-2'

W. FEDUSKA: 'An Evaluation of the Diffusion-Bonding Characteristics of Zircaloy-2.'

Welding Journal, 1959, vol. 38, July, pp. 289s-95s.

Because of its good resistance to corrosion by high-temperature water and its relative transparency to thermal neutrons, 'Zircaloy-2' (a zirconium-base alloy containing tin 1·5, iron 0·12, chromium 0·10, nickel 0·05, per cent.) has been used for fuel-element cladding in pressurised-water reactors. Attempts to produce strong, ductile and corrosion-resistant joints in the alloy by brazing with high-temperature brazing alloys have not been fully successful (see, for example, abstract in *Nickel Bulletin*, 1957, vol. 30, No. 9, pp. 176-7). Diffusion-bonding, however, appeared to offer a method of obtaining the required combination of properties in the joint, and an exploratory investigation of the diffusion-bonding characteristics of 'Zircaloy-2' was carried out by the author. The results of the investigation are reported in the present paper.

The diffuser elements selected for study were limited to those possessing relatively low thermal-neutron-absorption cross-sections and relatively small atomic radii. Powders of the following metals were employed: aluminium, chromium, copper, iron, manganese, nickel, nickel-phosphorus, silicon, tin, zinc.

'Zircaloy-2' plates $\frac{1}{8}$-in. (0·3-mm.) thick and 1-in. (2·5-cm.) square were used, in preliminary wettability tests, to evaluate the reactions involved in the 'Zircaloy-2'/diffuser-element system. Finally, to simulate actual diffusion-bonding conditions, diffusion tests were carried out with each of the diffuser elements, using sandwich specimens machined from 'Zircaloy-2' to dimensions which are given. After both the wettability and diffusion-bonding sandwich tests the joint/interface regions were subjected to microscopical examination, to ascertain the nature of the diffusion reactions.

From the results of the experiments the author concludes that diffusion-bonding offers promise as a means of joining 'Zircaloy-2' components.

The rates of diffusion of the diffuser elements into 'Zircaloy-2', determined for the duration of the diffusion-bonding test cycles, decreased in the following order: copper (0·0029 i.p.m.), nickel, nickel-phosphorus, silicon, manganese, iron, and chromium (0·0005 i.p.m.). No apparent diffusion into alpha-zirconium phase occurred when aluminium, tin, and zinc were used as diffuser elements.

Welding of Ferritic to Austenitic Steels: Review of Progress

J. CLASS: 'Study of Progress in the Development of Permanent Joints between Ferritic and Austenitic Steels.'

Mitteilungen der Vereinigung der Grosskesselbesitzer, 1959, No. 60, June, pp. 181-207.

The increasing use of austenitic-steel tubes in steam-boiler plant has focussed attention on methods of welding ferritic grades of steel to austenitic material to produce smooth joints which would withstand the high-temperature conditions characteristic of such service. This paper comprises a comprehensive survey of the metallurgical problems involved in welding the two steels, and of the various methods which have been proposed to overcome them. The review is supported by copious references to the literature (the *bibliography* contains 43 items), and the author's discussion is correlated throughout with numerous figures, photomicrographs, and tabular and graphical data. Most of the information relates to low-alloy chromium-molybdenum ferritic steels and to chromium-nickel austenitic steels of 16-13 or 18-8 type. The paper is divided into eleven sections, the scope of which is indicated below.

I. *Indirect Joints Between Ferritic and Austenitic Steel Tubes*

Examples of such joints are discussed.

II. *Problems Involved in Direct Welding of Ferritic and Austenitic Steels*

The two types of steel differ in their mechanical, physical and chemical properties (in particular, in coefficient of thermal expansion and thermal conductivity). The influence of these differences on welds between the two steels is discussed, and consideration is also given to the effects of thermal cycling during service. The advantages and limitations of electric-arc, flash-butt and pressure-welding techniques are briefly outlined, and attention is directed to the necessity, irrespective of the welding method, of ensuring that the weld joining austenitic and ferritic tubes is free from notches.

III. *Service Performance of Welded Austenite/Ferrite Joints*

Reference is made to literature reporting the behaviour of austenite/ferrite welds (or of joints in ferritic steel produced using austenitic weld metal) during service in turbines or steam plant. This information is discussed in relation to the light it throws on the stresses to which the joint is subjected during temperature cycling (*see also* abstract of paper by SCHILLER, p. 314).

IV. *Diffusion Reactions in the Transition Zone of the Weld.*

If suitable precautions are not taken, migration of carbon from the ferritic to the austenitic steel components of a welded tube can (as a result of welding, heat-treatment, or long-time exposure at high temperatures) occur in the transition zone of the weld. Literature and data are cited, in this connection, to illustrate the deleterious effects which result from decarburisation of the ferritic steel and the ensuing microstructural changes in the region of the weld. In particular, it is emphasised that the diffusion reaction is governed by temperature and time at temperature.

V. *Methods of Preventing Carbon Migration at High Temperatures*

Two methods are discussed: (1) introduction of

a thin diffusion barrier at the interface of the weld (e.g., of nickel in diffusion-welded overlapping joints); (2) use (as a junction between the ferritic and austenitic steels) of a ferritic steel with a carbon activity coefficient which more nearly approaches that of the austenitic steel. In the latter connexion reference is made to the successful use of various niobium-stabilised ferritic steels.

VI. *Risk of Stress Corrosion*

Reports of stress corrosion in austenite/ferrite joints are discussed. It is considered that resistance to such attack is increased by use of stabilised ferritic steels, and of high-nickel-alloy filler metal. No likelihood of stress corrosion exists in the absence of an aqueous corrosive medium.

VII. *Austenite/Ferrite Weld Junctions*

Mannesmann-type junctions are discussed. A detailed account of these junctions is given in the paper by LINDEN and HENNEKE: see following abstract.

VIII. *Use of Nickel-base Alloys in Welding Austenitic to Ferritic Steel*

The section covers the development of nickel-base alloys suitable for use as transition metal in welding austenitic to ferritic steel. The coefficients of thermal expansion of these alloys are adjusted so as to minimise the effects of the different thermal expansions of the two steels. Reference is made to work and data on several alloys (*inter alia*, a nickel-base alloy containing 3 per cent. chromium, and the nickel-molybdenum alloy 'Hastelloy W'), but the main part of the section is concerned with discussion of the composition and properties of the nickel-chromium - iron welding electrodes and wire designated 'Inco-Weld A'. Mention is also made of the advantages offered by nickel-base filler metal in minimising the effects of dilution with the basis metal, and in their excellent heat- and corrosion-resistance.

IX. *Investigation of Hardness and Structural Changes in the Transition Zone of the Weld*

To supplement the information given in Sections IV and VIII, the author presents, in some detail, the findings of studies of the effects of prolonged exposure, at 625° and 700°C., on welds joining ferritic and austenitic steel produced using the following types of weld metal: 18-8-niobium steel, an austenitic alloy containing 20 per cent. cobalt, 'Hastelloy W' and 'Inconel'. The results of metallographic examination, and of intergranular - corrosion, stress - corrosion, tensile and bend tests are presented.

X. *Tests on Welded Tubes*

The data tabulated were derived from microhardness, notched-bar impact-bending, tensile and bend tests on specimens taken from joints obtained using 'Inconel' electrodes to weld low- or high-alloy chromium-molybdenum ferritic steel to a niobium-stabilised vanadium-containing precipitation-hardenable austenitic steel. The welds were found to be sound and mechanical properties were high.

XI. *Investigation of the Effects of Temperature Cycling on the Life of Austenitic-Steel/Ferritic-Steel Joints*

The data presented relate to investigations (carried out, and previously reported, by TUCKER and EBERLE) involving thermal-cycling tension tests on specimens containing dissimilar welds. (See abstracts in *Nickel Bulletin*, 1951, vol. 24, No. 9-10, pp. 206-7; *ibid.*, 1957, vol. 30, No. 3, pp. 43-4.)

Factors deleterious to austenite/ferrite joints and methods of minimising their effects are summarised in a table which is reproduced on p. 313.

Pressure-Welded Austenite/Ferrite Tube Junctions

H. LINDEN and H. HENNEKE: 'Pressure - Welded Austenite/Ferrite Junctions of Mannesmann-type.' *Mitteilungen der Vereinigung der Grosskesselbesitzer*, 1958, No. 54, June, pp. 165-75.

The joining of ferritic steels to austenitic grades is one of the problems which, as a result of the increasing use of heat-resisting steels to withstand higher temperatures and pressures, confronts the engineer in the construction of steam-boiler plant. In this paper the author discusses the results of investigations designed to evaluate the properties of austenite/ferrite junctions which were developed (in the laboratories of Mannesmann A.G.) as a solution to this problem.

The tube junctions are fabricated from two tubes of the desired dimensions, one made in ferritic, the other in austenitic steel. The end of the austenitic-steel tube tapers gradually towards the inner surface of the tube wall; the end of the ferritic material tapers at the same angle towards the outer surface of the tube wall. The tapered ends of the tubes are fitted together (the austenitic inside the ferritic), pressure welded at an appropriate temperature, and the composite tube is then heat-treated and finally mechanically worked. This tube then serves as a junction which enables a ferritic- and an austenitic-steel tube to be joined together, without deleterious microstructural changes, by being welded to a like material at the appropriate end of the junction. (In junctions intended for service at temperatures higher than 550°C. a thin layer of nickel is interposed between the joint surface, to prevent carbon diffusion between the two steels.) For the austenitic end of the junction use is generally made of a niobium-stabilised chromium-nickel-(molybdenum) steel (e.g., of 16-13 type); the ferritic component is either of low-alloy chromium-molybdenum steel or of 12 per cent. chromium-molybdenum-vanadium steel. These same materials were used for the junction tubes tested in the present investigation.

The test programme involved:

(1) Tensile and bend tests (at room temperature and at temperatures in the range 500°-600°C., in

Factors Deleterious to Welds Between Tubes of Austenitic and Ferritic Steels and Methods of Minimising their Effects

(*See abstract on p. 312.*)

Factor Involved	Preventive Measure	Method Suggested
1. Carbon Migration	Stabilisation of the ferritic steel by addition of carbide formers	Use of a niobium-stabilised steel
	Use of an intermediate junction or layer of a stabilised nickel-rich alloy (chromium content approximately similar to that of the ferritic steel)	
	Use of steels which do not differ greatly in their activity coefficients	
	Use of a barrier layer of nickel	Use of pressure-welded junctions with overlapping weld faces
	Minimising diffusion during production of the weld; annealing, if essential after welding, only at low temperatures	
2. Differences in the thermal expansion of the two steels	Use of weld metal with suitable thermal-expansion properties (similar to that of the ferritic steel or at least less than that of the austenitic steel)	Use of special electrodes of high nickel content
3. Stressing as a result of fluctuations in temperature	Modification of the operational conditions. Maintaining the temperature of the joint during shut-down by means of special heating apparatus	Electric heating muffle
	Whenever possible, machining the inner and outer surfaces of the tube	
4. Stress corrosion	Ensuring that the joint is dry (outside the wet-steam zone); stress-relief treatment of the weld	
5. Risk of rupture	Use of bevelled, conical joint faces	Overlapping joint faces
6. Insufficient control over performance during service	Bevelled, conical joint faces permit use of ultrasonic-inspection techniques	Junctions with overlapping joint faces
7. Corrosion of the decarburised zone of the weld on the gas side	Overwelding with corrosion- and heat-resisting weld metal of suitable thermal expansion characteristics	(80-20 nickel-chromium alloy)

some cases after 1000 hours' exposure at 550° or 600°C.) on specimens machined from tube junctions, with or without intermediate nickel layers, in a longitudinal direction.

(2) Tensile tests, at room temperature (in some cases after long-time exposure at 500°, 550° or 600°C.) and at 550° and 600°C., on specimens machined perpendicularly to the weld face.

(3) Creep tests on longitudinal specimens.

(4) Creep tests on complete junction tubes.

(5) Tests to determine resistance to temperature cycling.

(6) Metallographic examination of specimens heated for up to 5,000 hours at temperatures in the range 500°–650°C., carried out to determine the influence of temperature, and time at temperature, on the structure of the austenite/ferrite transition zone of the junctions.

(7) Tests, in hot magnesium-chloride solutions, to determine susceptibility to stress corrosion.

(8) Ultrasonic testing of junctions.

From the results of the investigation the authors conclude that it is possible by pressure welding to obtain a sound joint between the austenitic and

ferritic components of the junction, and that the soundness is unaffected even by long-time stressing at high temperatures. This conclusion is applicable, in particular, to junctions which incorporate a thin diffusion barrier of nickel at the weld surface.

Severe thermal-shock tests, designed to take account of the difference in the thermal expansions of the two component materials, caused no rupture of the joint.

The results of the metallographic study of the effects of carbon migration from the ferrite to the austenite, and of the inhibiting effect of an intermediate layer of nickel, emphasise the value of interposing such a layer at the weld face. In view of carbon diffusion at temperatures above 550°C., heat-treatments should be as short as possible, but experience has shown that even if, as a result of short-time heat-treatment, decarburisation occurs in a narrow zone of the junction, any deleterious effect is counteracted by the fact that the surface of the weld is six to ten times larger in area than the cross section of the tube.

Using an equation reported in the literature, the authors show that in junctions without an intermediate nickel layer the structural changes caused by carbon diffusion can be approximately predicted as a function of time and temperature.

No stress-corrosion cracking occurred in the junctions despite the severity of the corrosion test, a result which is attributed to the cathodic protection conferred on the austenite by the ferrite.

Ultrasonic-test techniques could be successfully applied to determine the soundness of the welds, and evaluation by such techniques showed good agreement with the results of subsequent metallographic examinations.

See also

Thermal-Shock Testing of Pressure-Welded Austenite/Ferrite Junctions

R. SCHINN and W. RUTTMANN: 'Thermal-Shock Tests on Austenite/Ferrite Junctions.'

Mitteilungen der Vereinigung der Grosskesselbesitzer, 1958, No. 57, Dec., pp. 407–11.

The tests were carried out on four types of pressure-welded austenite/ferrite junction tubes ranging from 38 to 140 mm. and from 6 to 15 mm. in, respectively, diameter and wall thickness. The junctions were welded from niobium - stabilised 16–13-type chrom - ium-nickel-(molybdenum) austenitic steel and low-alloy chromium-molybdenum or 12 per cent. chromium-molybdenum-vanadium ferritic steel. To prevent carbon migration a layer of nickel was interposed at the joint interface.

The junctions were cycled between 570°C. and 300°C. (or, in some cases, 50° or 160°C.), using flowing water as a quenching medium. Such temperature cycling was far more severe than that which could be expected in service (i.e., as junctions between ferritic valve bodies and austenitic pipelines carrying live steam), and were intended to determine only whether the pressure weld was the weakest part of the junction.

After testing, the junctions were subjected to metallographic examination. Photomicrographs included in the paper show that the thermal stressing had been sufficiently severe to induce transcrystalline cracking in the basis metal, but that little or no damage had been caused to the joints of three of the types of junction studied. In the fourth type the joint had separated in the nickel layer.

Data derived from creep tests at 600°C., on test pieces machined from two of the junctions, showed that the creep-resistance of both junctions was similar to, or higher than, that of the 12 per cent. chromium - molybdenum - vanadium steel which formed one of the component materials.

Stresses at the Joint Face of Austenite/Ferrite Welds

A. SCHILLER: 'Stresses at the Joint Face of Two Tube Materials with Different Coefficients of Thermal Expansion and Moduli of Elasticity.'

Mitteilungen der Vereinigung der Grosskesselbesitzer, 1958, No. 54, June, pp. 176–80.

Formulae are presented which permit calculation of the internal stresses occurring at the joint face of austenitic-steel/ferritic-steel welds due to differences in the thermal expansion of the two steels. The study relates to tubes joined either by butt welds or by overlapping (Mannesmann-type) welds. It is shown that the stresses decrease with increase in temperature, and are at a maximum at room temperature.

Welding of Dissimilar Metals with 'Inco-Weld A' Electrodes and Wires

'Welding of Dissimilar Metals.'

Welding Engineer, 1959, vol. 44, Mid-June, pp. 5–10, 12–13.

In 1958 The International Nickel Company, Inc., issued a report giving details of laboratory and field experience with the nickel-chromium-iron welding electrodes and wire, designated 'Inco-Weld A', which were developed by the Company specifically for welding dissimilar materials. The main purpose of that report was to present data on typical properties of welds so produced. (The scope of the report, and the compositions of both electrodes and wire, are given in the pertinent abstract in *Nickel Bulletin*, 1958, vol. 31, No. 12, pp. 350–1.) In the present article the same data on properties are reproduced, but the emphasis is on welding practice.

'Inco-Weld A' has been used in a wide range of production and maintenance applications to produce sound, reproducible welds in various combinations of dissimilar metals, e.g., mild steels, low-alloy steels, stainless steels and high-nickel alloys: applications described in the article illustrate the range of metals successfully welded, and the versatility of the electrodes and wires. The major part of the article is, however, devoted to a discussion of the precautions which must be taken in welding procedure (both in metal-arc inert-gas and tungsten inert-gas welding) to obtain optimum results. Factors on

which information is given include joint preparation for various thicknesses of metal, amperages recommended for down-hand, vertical and overhead welding, selection of electrode or wire diameters, and arc control.

Data on the weldability of dissimilar metals (using resistance- or gas-welding techniques) are summarised in tables.

ANALYSIS

Magnetometric Titration of Nickel

See abstract on p. 280.

PATENTS

Catalytic Conversion of Hydrocarbons

In the cyclic catalytic conversion of hydrocarbons to a combustible gas the surface of the refractory material forming the heat-storage zone is lined with a film of nickel and/or cobalt. The coating is stated to prevent or minimise premature thermal cracking of the reactant hydrocarbon gas as it is passed through the refractory zone while being heated to the temperature required for subsequent catalytic conversion. The preferred lining material is nickel, which may be sprayed onto the refractory in the form of a water-soluble nickel compound (for example, $Ni(NO_3)_2.6H_2O$), which, by subsequent oxidising and reducing treatment, is converted to elemental nickel.

C. G. MILBOURNE, assignor to UNITED GAS IMPROVEMENT CO. *U.S. Pat.* 2,868,632

Protection of Elastomers from the Effects of Sunlight and Atmospheric Ozone

Olefinic elastomers, in the solid or latex form, are protected from the deleterious effects of sunlight and atmospheric ozone by incorporating into the elastomer, at the end of the mixing cycle, from 0·1 to 5 per cent. (preferably 1-2 per cent.) of a nickel xanthate (alkyl-xanthate, alicyclic xanthate, aryl-xanthate or aryl-alkyl-xanthate). Titanium oxide, at least one micro-crystalline wax and an amorphous paraffin, and an anti-ozone substance selected from the following may also be added to the elastomer: diphenyl - p - phenylene - diamine, zinc mercapto - benzimid-azolate and dinaphtyl-para-diphenylene-diamine.

P. I. KARMITZ and C. P. PINAZZI, assignors to MANUFACTURES DE PRODUITS CHIMIQUES DU NORD ETABLISSEMENTS KUHLMANN. *Canad. Pat.* 579,216

Improvements in Electroless Nickel Plating

Nickel is deposited from an aqueous solution containing a nickel salt and an alkaline hypophosphite. The solution, operated at a temperature of 95°C., is kept slightly acidic by periodical additions of a base, and the improvement consists in adding nickel and hypophosphite in amounts, proportional to the amount of base used, which ensure that the ratio of the molecular concentrations of nickel and hypophosphite is maintained constant.

G. LALLEMENT. *French Pat.* 1,179,734

Nickel Plating of Uranium

A protective coating is applied to uranium by a procedure involving the initial stages of (1) anodically etching the uranium in an aqueous solution (preferably of phosphoric or hydrochloric acid) containing chloride ions; (2) cleaning the etched uranium in an aqueous nitric-acid solution; (3) promptly nickel plating from a nickel-sulphate solution. The nickel coating, serving as a diffusion barrier, then permits application of a second protective coating which would otherwise be unsuitable because of diffusion or alloying with the basis metal. To provide the second coating the nickel-plated uranium component is preferably dipped in aluminium or a low-melting aluminium-silicon alloy, or, alternatively, is immersed in a molten bath of sodium chloride and/or potassium chloride and/or lithium chloride at 700°-800°C. for a sufficient time to alloy the nickel with the uranium.

A. G. GRAY, assignor to U.S. ATOMIC ENERGY COMMISSION. *U.S. Pat.* 2,894,884

Gas Plating of Fibres

Materials (such as fibres, filaments, fabrics, tapes, etc.) which are not heat-conductive and which soften at a relatively low temperature are difficult to metallise by gas plating, since most thermally decomposable metallic compounds require to be used at a higher temperature than that at which the materials soften. According to the claims of the invention, such materials are successfully plated by passing the heated strand of material longitudinally through a swirling atmosphere of a thermally decomposable metal-bearing compound at a relatively high speed. The swirling atmosphere provides for rapid removal of gases of decomposition which otherwise would tend to obstruct the ingress of the plating gas.

The process is exemplified by a description of the procedures used to nickel plate glass and nylon fibres by decomposition of nickel carbonyl, and to iron plate 'Orlon' by decomposition of iron penta-carbonyl. In the case of the nylon, the filaments were heated to about 275°F. (135°C.) and passed through the nickel-carbonyl atmosphere at a speed of about 20 ft./minute (6 m./minute).

H. R. NACK, assignor to COMMONWEALTH ENGINEERING COMPANY OF OHIO. *U.S. Pat.* 2,887,088

Low-Melting-Point Plutonium-Nickel Alloys

The alloys covered by the patent, developed for use as liquid-metal fuels in neutronic reactors, possess the low melting points (i.e., about 200C°. lower than that of pure plutonium) and neutronic characteristics prerequisite for such use. Limits of composition are: iron and/or cobalt and/or nickel 8 - 15 at. per cent., plutonium remainder.

Three preferred plutonium-base compositions contain nickel 14, iron 9·5 and cobalt 12, at. per cent., respectively.

W. CHYNOWETH, assignor to UNITED STATES ATOMIC ENERGY COMMISSION. *U.S. Pat.* 2,890,954

Production of Acicular Metal Particles from Magnetic-Metal Carbonyls

Acicular magnetic-metal or magnetic-alloy particles are produced by thermal decomposition of a magnetic-metal carbonyl (or of a mixture of metal carbonyls containing at least one magnetic-metal carbonyl), in the presence of a substantial, controlled, local, unidirectional magnetic field, to cause aggregation of the decomposition product along lines of magnetic flux. The apparatus employed to carry out the invention is described.

It is stated in the preamble that the particles have length/diameter ratios in the range 10–100 and that lengths vary from several millimeters down to micron dimensions. The size of the particles is a function of the reaction rate, temperature of the reaction and concentration of the carbonyl vapours; the length/diameter ratio is a function of the flux density of the magnetic field across the reaction zone.

Fibres of ferromagnetic materials are readily obtained by decomposition of a mixture of the appropriate carbonyls: for example, particles of nickel steel, 'Permalloy' or 'Perminvar' may be obtained from mixtures of the carbonyls of iron, nickel and cobalt.

R. W. FABIAN and J. L. SIENCZYK, assignors to BUDD CO. *U.S. Pat.* 2,884,319

Air-Hardening Graphitic Steel

The patent covers steels which, when hot-worked and air-cooled from an austenitising temperature, possess a microstructure characterised by a high-strength matrix throughout which are uniformly distributed spheroidised carbides and a minimum of 0·2 per cent. graphitic carbon, and exhibit a hardness of at least Rockwell C 58 throughout sections at least 6 in. (15 cm.) in diameter. Machinability and tempering characteristics are stated to be good.

The following limits of composition are claimed: carbon 1–2, silicon 1–1·5, chromium 0·2 max., manganese 1·7–2·5, molybdenum 1·3–1·75, nickel 1–2·5, per cent.

C. F. JATCZAK, assignor to TIMKEN ROLLER BEARING CO. *U.S. Pat.* 2,883,281

Attachable Hard-Surfacing Component

Materials such as rubber, leather, zinc, brass or aluminium are hard-surfaced by attachment of a wear-resistant element conforming to the configuration and dimensions of the area to be hard-surfaced. The element comprises a perforated metallic support: the outer face is sprayed with a hard metallic powder which is then fused; the inner face remains relatively soft and is bonded to the material to be hard-surfaced. The bond may be effected by such methods as soldering, brazing or use of adhesives.

In an example cited a nickel-chromium-alloy wire cloth is sprayed with nickel-chromium-boron-alloy powder which is then fused in a furnace to produce a surface hardness of Rockwell C 58.

S. LOW. *Brit. Pat.* 817,410

Production of Nickel-containing Alloy Powders

The invention comprises a method of producing homogeneous compositions, in particular, alloy powders, cermet powders, pure alloys in regulus form, alloys of materials difficult to reduce, minerals, synthetic minerals and ceramics.

A homogeneous solution of the appropriate constituent elements is prepared in a volatile solvent (such as nitric acid, sulphuric acid, ammonia or water). The solution is then rapidly dried under conditions which ensure that the volatile solvents are driven off but preclude segregation of the constituent elements. The rapid drying has the effect of forming a homogeneous molecular mixture of the elements in solid form, and this mixture may then be further treated to obtain a regulus alloy or a molecular mixture in powder form.

Alloy powders which can be processed according to the invention include those which can be reduced from a mixed oxide below the melting temperature of the alloy and below the temperature at which one or more of the alloy constituents melt and segregate from the remaining unreduced oxides. Examples are: 'Stellite' alloys, brass, bronze, nickel silver, silver-nickel alloys, steels (including stainless and tool steels), 'Nichrome' and 'Monel'. Among the materials for which details of procedure are given in the preamble are: 18-8 steel (in powder or regulus form), 18-8 steel/Al_2O_3 cermet, and 'Monel' powder. The 'Monel' powder is produced by dissolving appropriate amounts of nickel, copper and iron turnings in nitric acid, heating to drive off the solvents, reducing the solid product in a hydrogen atmosphere, and finally grinding to the desired particle size.

B. H. TRIFFLEMAN. *U.S. Pat.* 2,893,859

Precipitation-Hardenable Stainless Steel

The inventors have established that the addition of nitrogen to precipitation-hardenable stainless steels containing a precipitation-hardening element such as aluminium prevents hardening as a result of solution-treatment, and renders the steel uniformly responsive to transforming and precipitation-hardening treatments. The steel claimed is characterised by a hardness value of less than 95 R_b after solution-annealing and air-cooling (when the structure is substantially austenitic), and by a hardness value of at least R_c 38 after a double thermal treatment to achieve phase transformation (to martensite) and age-hardening. Limits of composition are: carbon 0·05–0·08, chromium 16–17·5, nickel 5·5–6·75, aluminium 0·9–1·8, nitrogen 0·12–0·18, per cent.

C. S. WALTON and W. F. WHITE, assignors to UNITED STATES STEEL CORPN. *U.S. Pat.* 2,892,702

Heat-Resisting Austenitic Steel

The heat-resisting stable single-phase austenitic steel of the invention falls within the following limits of composition: carbon 0·15–0·4, chromium 19·5–24, nickel 4 min., manganese 4 min. (nickel + manganese 8–13), copper 0·5–3, nitrogen 0·15 min., per cent. Compared with steels of the same composition but without copper and with a carbon content greater than 0·4 per cent., the steels covered exhibit greatly improved stress-rupture characteristics: an improvement of 30 per cent. in rupture life at 1200°F. (650°C.), under a stress of 50,000 p.s.i. (22·5 t.s.i.; 35 kg./mm².), is specifically claimed.

W. R. KEGERISE and B. T. LANPHIER, assignors to CARPENTER STEEL CO. *U.S. Pat.* 2,891,858

Nickel-Vanadium-base High-Temperature Alloy

A high-temperature alloy, claimed to exhibit improved heat-resistance at temperatures higher than 500°C., contains vanadium 15–30 per cent., cobalt and/or nickel remainder. This composition may be modified by replacing up to 10 per cent. of the vanadium, nickel or cobalt contents by aluminium, titanium, chromium, tantalum, iron, niobium, manganese, zirconium, molybdenum, silicon or tungsten, and by additions of up to 1 per cent. cerium, up to 0·5 per cent. boron, and up to 0·5 per cent. carbon and 0·5 per cent. nitrogen.

The alloy is heat-treated by rapid cooling from a temperature above 1000°C., followed by annealing in the range 400°–1000°C. (preferably in the range 400°–750°C.) to obtain optimum hardness. The properties of the alloy are not dependent upon a precipitation-hardening mechanism, and hardness values are therefore governed solely by the temperature of heat-treatment and are independent of time at temperature. In a diagram contained in the patent, the Vickers hardness exhibited by an alloy containing cobalt 38–75, nickel 38–75, vanadium 22·5, per cent., is correlated with annealing temperatures.

DEUTSCHE EDELSTAHLWERKE A.G. (inventors, W. KÖSTER and F. SPERNER). *German Pat.* 1,061,520

Nickel-Chromium-Iron Heat-Resisting Alloy

The nickel-chromium-iron alloy to which the patent relates possesses a tensile strength at 1400°F. (760°C.) greater than 50,000 p.s.i. (22·5 t.s.i.; 35 kg./mm.²), an oxidation rate in air of less than 0·01 in./year (0·25 mm./year) at 1800°F. (980°C.), and a stress-rupture life of about 1100 hours at a stress of 7000 p.s.i. (3 t.s.i.; 5 kg./mm.²) at 1600°F. (870°C.). Limits of composition are as follows: nickel 10–25, chromium 25 max. (nickel + chromium 25 – 50), manganese 5–15, silicon 0·9–1·5, niobium 0·75–1·5, nitrogen 0·05–0·5, carbon 0·3–0·5, sulphur 0·05 max., phosphorus 0·05 max., per cent., remainder iron.

W. F. FURMAN and H. T. HARRISON, assignors to DURALOY CO. *U.S. Pat.* 2,892,703

Electric Heating Device for Service above 870°C.

An improved electric heating device consists of a heating element, coated with a layer of 0·00025–0·005-in. (0·006–0·125-mm.) of nickel, which is embedded in a mass of magnesium oxide (or a mixture of magnesium oxide with another electrically-insulating, but thermally-conductive, material) surrounded by a sheath of a nickel-chromium-iron alloy. The inner surface of the sheath is preferably also coated with nickel. For the heating element use is made of an alloy containing iron and/or nickel and/or cobalt 65, chromium and/or molybdenum 10–35, per cent., with or without additions of up to 20 per cent. of such elements as tungsten, niobium, zirconium, titanium, silicon, manganese, aluminium.

Existing devices of this type tend to fail at temperatures higher than 870°C., due to the adverse effects, on the insulating properties and dielectric strength of the refractory material, of migration of oxides from the heating element into the insulation. The device claimed has an increased service life at temperatures in the range 870°–1200°C., an improvement which is attributed to the protection against oxidation conferred on the heating element by the nickel layer.

MOND NICKEL CO., LTD. *Brit. Pat.* 815,312

Passivation of Stainless Steels

The invention relates to a method of passivating stainless steels so as to render them non-reactive to hydrogen peroxide. Passivation effected by present methods is not of satisfactory duration, and, with respect to some steels (e.g., A.I.S.I. Type 303 18-8 chromium-nickel steel), these methods are stated to be completely unsatisfactory.

According to the claims of the patent the stainless steel is immersed in a pickling bath, heated at 700°–800°F. (370°–425°C.) in an oxidising atmosphere, and finally exposed to hydrogen peroxide until all reaction ceases. The following application of the method to passivation of Type 303 stainless steel is specifically claimed: immersion in 20 per cent. caustic-soda solution at room temperature for about 30 minutes; rinse; immersion in 50 per cent. nitric acid at room temperature for about 24 hours; rinse; immersion in 10 per cent. chromic-acid solution at room-temperature for 1 hour; rinse; immersion for 1 hour in a boiling sodium-picrate solution containing about 2 per cent. picric acid and an excess of sodium hydroxide; rinse; heating at 750°F. (400°C.) for about 2 hours in an oxidising atmosphere; cool; immersion in a 30 per cent. solution of hydrogen peroxide until all reaction ceases.

C. F. CARRIGAN, assignor to FAIRCHILD ENGINE AND AIRPLANE CORPN. *U.S. Pat.* 2,890,974

Protection of Steel Tubing from Hydrogen Embrittlement and Stress-Corrosion Cracking

Steel oil- or gas-well tubing is protected from hydrogen embrittlement or stress-corrosion cracking (occurring as a result of contact with atomic or nascent hydrogen formed in the well) by incorporation within the tubing of a lining of a metal or alloy pervious to atomic hydrogen. The lining is fitted tightly within, but not bonded to the surface of, the tubing, enabling any atomic hydrogen that may form inside the liner tube to diffuse through to

its outer surface, where the atoms preferentially combine to form molecular hydrogen. This molecular hydrogen, which is substantially inert to the steel tubing at the temperatures obtaining in the wells, collects between the liner and the tubing.

The outer shell of the composite tubing is preferably made of a high-strength steel containing chromium 0·75–1, molybdenum 0·08–0·1, manganese 0·5–1·25, nickel 0·75–1·25, per cent. The liner, which is inserted into the outer shell of the tubing and expanded by hydrostatic pressure, consists of nickel, copper or a nickel- or copper-base alloy. Preferred compositions are: nickel 63–70, copper 25–30, iron 0–2, manganese 0–2, aluminium 0–4, silicon 0–1, carbon 0–0·3, per cent.; nickel 50 min., chromium 10–35, per cent., remainder iron.

MOND NICKEL CO., LTD. *Brit. Pat.* 819,618

Stainless Steel Resistant to Corrosion by Nitric Acid

Stainless steels free from delta ferrite and falling within the following composition range are claimed to exhibit a corrosion rate of less than 0·05 mm./month when exposed, after sensitisation for 2 hours at 677°C., to 65 per cent. boiling nitric-acid: carbon 0–0·03, silicon 0–0·6, manganese 0–2, nickel 14·3 min., chromium 17 min., molybdenum 2–3·1, per cent. The maximum manganese, nickel and chromium contents are preferably 1·5, 16 and 20, per cent., respectively. The amount of silicon present should be as low as possible.

UNITED STATES STEEL CORPN. (inventors, R. SMITH and C. O. TARR). *French Pat.* 1,184,404

Chromium-Nickel Stainless Steels Resistant to Pitting Corrosion

The patent covers the powder-metallurgical production of chromium- or chromium-nickel-steel components (e.g., tubing, sheet, etc.) claimed to be resistant to pitting corrosion in solutions containing halogen ions. Resistance to pitting is conferred by adding to the powderised steel small amounts of metal powders exhibiting a lower electrochemical potential in halogenated solutions than that of the steel powder. This powder mixture is then compacted into the desired form and sintered.

The steel powder is produced by atomising a stream of molten steel in contact with a jet of water under high pressure. The silver or copper which comprise the addition metals are added to the steel powder either as powders or in the form of aqueous solutions of their nitrates, the silver in amounts of 0·05–2 per cent. (preferably 0·3 per cent.), the copper in amounts of 3–5 per cent. (preferably 4·5 per cent.).

MANNESMANN A. G. *French Pat.* 1,178,577

Heat-Resistant Copper-base Brazing Alloy

A malleable brazing alloy having high strength and creep-resistance at elevated temperatures, and a melting point (about 1900°F. : 1035°C.) lower than that of pure copper, contains nickel 3·5–10·5, chromium 0·825–2·44, boron 0·175–0·56, per cent., remainder copper. The following preferred composition is claimed: copper 90, nickel 7, chromium 2, boron 0·3, per cent. The alloy is stated to exhibit high creep- and corrosion-resistance at temperatures in the range 900°–1200°F. (480°–650°C.) and to be suitable for such applications as brazing gas-turbine components.

T. L. WOOLARD. *U.S. Pat.* 2,891,860

Weldable Heat-Treatable Stainless Steel

The steel to which the patent relates is characterised, when welded, by (1) a martensitic microstructure in the region of the weld substantially free from retained austenite and containing no more than about 5 per cent. ferrite, and (2) by a maximum hardness of Rockwell C 38 in the heat-affected zone. The steel, which is stated to be readily heat-treated and to have satisfactory strength at elevated temperatures and improved resistance to weld cracking, contains carbon 0·02–0·06, nitrogen 0·01–0·04 (carbon + nitrogen 0·08 max.), silicon 0·4 max., chromium 9–14, manganese 6 max., nickel 4 max., per cent. The minimum nickel and manganese contents are defined (in relation to the chromium, carbon and nitrogen contents) in a diagram included in the patent. A preferred composition comprises: carbon 0·03, silicon 0·4 max., nitrogen 0·02, chromium 11–12, nickel 1·5, manganese 0·5, per cent.

W. R. KEGERISE, assignor to CARPENTER STEEL CO. *U.S. Pat.* 2,891,859

Production of Non-Magnetic Stainless-Steel Welds

A submerged-arc-welding electrode wire for the production of non-magnetic crack-resistant stainless-steel welds contains chromium 12–30, nickel 7–35, manganese 14·7–23·1, silicon 0–3, molybdenum 0–4, copper 0–5, niobium/tantalum 0–1·5, titanium 0–1·5, per cent., remainder iron.

G. E. LINNERT and R. M. LARRIMORE, assignors to ARMCO STEEL CORPN. *U.S. Pat.* 2,894,833

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks.*

THE MOND NICKEL COMPANY LIMITED
THAMES HOUSE · MILLBANK · LONDON, S.W.1

PRINTED IN ENGLAND *by* WIGHTMAN MOUNTAIN LTD.
WESTMINSTER, LONDON, S.W.1